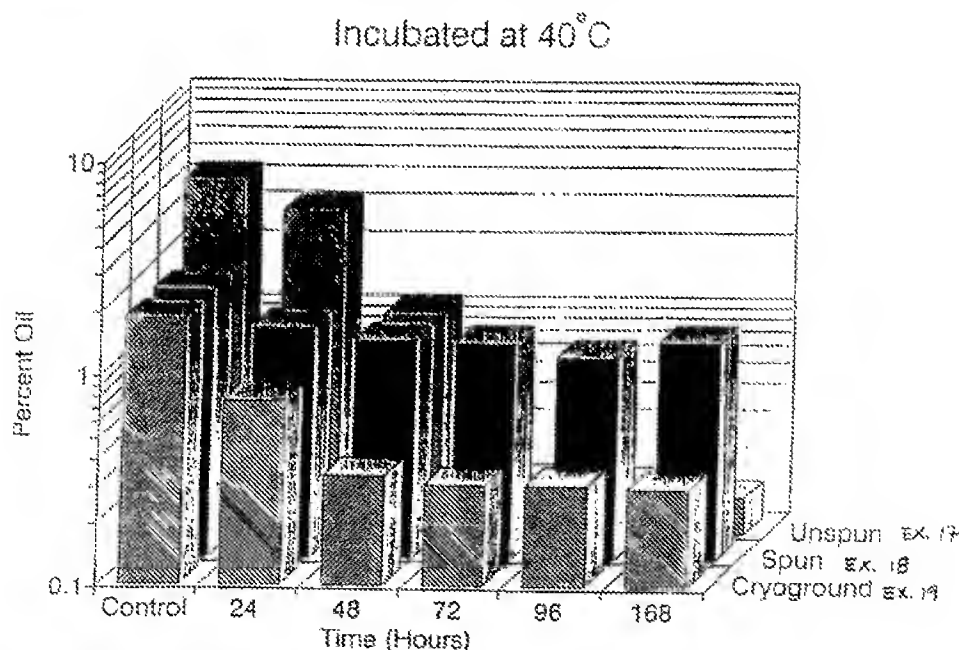




## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>5</sup> : A23G 3/30, A23L 1/222 A23P 1/04		A1	(11) International Publication Number: <b>WO 93/08699</b> (43) International Publication Date: 13 May 1993 (13.05.93)
(21) International Application Number: PCT/US92/09447 (22) International Filing Date: 30 October 1992 (30.10.92) (30) Priority data: 07/787,245 4 November 1991 (04.11.91) US (60) Parent Application or Grant (63) Related by Continuation US 07/787,245 (CIP) Filed on 4 November 1991 (04.11.91) (71) Applicant (for all designated States except US): FUISZ TECHNOLOGIES LTD. [US/US]; 3818 Concorde Parkway, Suite 100, Chantilly, VA 22021 (US).		(72) Inventor; and (73) Inventor/Applicant (for US only): FUISZ, Richard, C. [US/ US]; 9320 Cornwell Farm Road, Great Falls, VA 22066 (US). (74) Agents: BARON, Ronald et al.; Hoffmann & Baron, 350 Jericho Turnpike, Jericho, NY 11753 (US). (81) Designated States: AU, BR, CA, CS, FI, HU, JP, KR, NO, PL, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, SE), OAPI patent (BF, BI, CF, CG, CI, CM, GA, GN, ML, MR, SN, TD, TG). Published With international search report.	

(54) Title: WATER-SOLUBLE DELIVERY SYSTEMS FOR HYDROPHOBIC LIQUIDS



## (57) Abstract

A solid delivery system for rapid release of hydrophobic liquids such as oleaginous materials, flavor oils, mineral oil and the like comprising a water-soluble flash-flow-formed matrix containing a micronized dispersion of a substantially hydrophobic liquid.

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WATER-SOLUBLE DELIVERY SYSTEMS FOR HYDROPHOBIC LIQUIDSCROSS-REFERENCED TO RELATED APPLICATIONS

The present invention is a continuation-in-part of Ser. No. 787,245 filed November 4, 1991, which in turn is a continuation-in-part of Ser. No. 602,485 filed October 24, 1990 now U.S. Patent No. 5,096,492, which in turn is a division of Ser. No. 283,742 filed December 13, 1988 now U.S. Patent No. 5,001,532, which is a continuation-in-part of Ser. No. 169,838 filed March 18, 1988 now U.S. Patent No. 4,855,326, which is a continuation-in-part of Ser. No. 040,371, filed April 20, 1987 now abandoned.

BACKGROUND OF THE INVENTION

The present invention is directed to a novel method of encapsulating finely divided or micronized, substantially hydrophobic fluids using flash-flow, e.g. melt-spun techniques, to produce delivery systems for use in food products and other comestibles, pharmaceuticals, gum and confectionery products, cosmetics and personal hygiene products. More particularly, the present invention relates to a delivery system having a solid, hydrophilic encapsulating material capable of undergoing those changes associated with the flash-flow phenomena occurring during, for example, melt spinning and having a multitude of finely divided or micronized hydrophobic oil dispersed therein.

The use of hydrophobic oils as flavorants or fragrances in comestibles, pharmaceuticals, cosmetics and the like has focused on ways to control the organoleptic impact either by delaying or accelerating the intensity of perception. In conventional comestible products such as foods, pharmaceuticals, gum and confectioneries, flavor oils have been added in the free state, as well as in the encapsulated form for the combined effects of immediate and delayed flavor perception. For example, U.S. Patent

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No. 4,485,118 to Carrol, et al. discloses a chewing gum composition which contains sequentially released plural flavor system. One of the flavors is encapsulated with a water-insoluble coating for delayed release and a separate  
5 flavor is introduced in free, uncoated form for immediate release. U.S. Patent No. 3,962,463 to Witzel discloses a chewing gum having a reduced content of flavor ingredients, obtained by impregnating or depositing solid  
10 flavor particles, such as microencapsulated flavor particles sorbed on an edible substrate, and placing them on the surface of the gum.

In the comestible art, encapsulation and coating techniques have also focused on protecting the flavor oils from reacting with other co-mixed chemicals, or from  
15 oxidation, evaporation or volatilization through direct exposure to the environment. Flavor oils have been combined with a variety of sweeteners, particularly, for example, in gum and confectionery products. Flavor oils are often aldehyde, ketone and ester compounds which are  
20 highly reactive with a host of other common materials found in comestible products, as well as being sensitive to heat. For example, one such material commonly added to comestibles which reacts quickly to lose its sweetness in the presence of flavor oils is aspartame. The result is a  
25 comestible product which lacks both flavor and sweetness and therefore suffers from lack of overall organoleptic quality and shelf-life instability.

Methods of encapsulating or coating oils have conventionally involved using matrices of other  
30 hydrophobic materials, such as melted and solidified fats and waxes, polymers such as polyvinyl acetate and solvents, and/or elastomeric materials. Simple mixtures of these hydrophobic matrices and the flavor and/or sweetener materials were prepared using solvents and/or  
35 heat to form a melt in order to incorporate the flavor oil

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into the matrix. Incorporation in the melted stage was required to obtain as much homogeneity and encapsulation as possible. The molten mass was then cooled to solidification and ground into particles.

5 U.S. Patent No. 4,740,376 to Yang discloses use of a melted high molecular weight polyvinylacetate blended with a hydrophobic plasticizer and a flavoring ingredient. The melt blend is cooled, ground into a particulate and incorporated into an edible product. The hydrophobic  
10 plasticizers employed are mono-, di- and triglycerides having a fatty acid chain length of 16 to 22 carbons.

U.S. Patent No. 4,722,845 to Cherukuri, et al. discloses stable cinnamon flavored chewing gum compositions comprising gum base, sweetener and a  
15 sweetener delivery system comprising a dipeptide or amino acid sweetener in a mixture of fat and high melting point (106°C) polyethylene wax. U.S. Patent No. 4,803,022 also to Cherukuri, et al. discloses a powdered flavor composition encapsulated in a hydrophobic matrix of fat or  
20 wax and containing thaumatin, monellin or dihydrochalcones as the sweetener.

U.S. Patent No. 4,824,681 to Schobel, et al. discloses an encapsulated sweetening agent which is protected from moisture and provides controlled release  
25 wherein the sweetening agent is encapsulated with a hydrophobic polymer and a hydrophobic plasticizer. Hydrophobic coating is also described in detail in U.S. Patent No. 4,828,857 to Sharma, et al. wherein a delivery system is disclosed having as a core material a sweetener  
30 or flavoring ingredient and a protective matrix formed by a fluidized bed spray coating.

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These processes involving the formation of molten hydrophobic mixtures have inherent disadvantages which include loss of volatile flavor components during the heating process and significant mixing requirements to ensure homogeneity. Separate grinding steps followed, which also generated vaporization of flavor components and, in the case of certain sensitive sweeteners, degradation and loss of sweetness. Additionally, simple mixtures have failed to provide adequate uniform coating protection to keep the core flavors and sweeteners in a sustained stabilized state. Other processes, in an attempt to improve over simple mixing techniques, have included spray congealing and fluidized bed spray coating. While these methods may overcome certain of the above-mentioned disadvantages, they still employ significant amounts of heat and/or solvents and plasticizers and do not result in micronized discrete flavor droplets within a matrix, but rather a non-uniform agglomeration of the flavor and/or sweetener with the hydrophobic carrier. See, for example, U.S. Patent No. 4,722,845 to Cherukuri, et al. Coating of sweeteners and oils with hydrophobic materials also interferes with the immediate release properties and up-front, instantaneous flavor and/or sweetness impact.

The delivery systems of the present invention represents a departure from conventional methods and their resultant products in a number of important ways. To begin with, instead of using hydrophobic encapsulating matrix materials, flash-flowable hydrophilic materials are employed. These materials must be capable of undergoing flash-flow processes such as melt-spinning without significant degradation or burning. Therefore, instead of simple mixing and grinding, spray congealing and spray coating techniques, flash-flow processing as later defined herein, is employed. The result is a solid particulate which constitutes a highly water-soluble, flash-flowable

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matrix or encapsulant in which there is dispersed throughout fine, micronized liquid droplets. During the flash-flow process, the encapsulant is exposed to extremely limited conditions of heat and then usually only for a fraction of a second. This substantially lessens the potential for volatilization of certain hydrophobic liquid components. In the case of flavor oils, analytical testing has demonstrated that the intensive delivery systems retain more flavor components than those processes of the prior art.

#### SUMMARY OF THE INVENTION

The present invention concerns a method of producing encapsulated hydrophobic fluids, and the resultant delivery systems made therefrom. The oils are contained in and protected by encapsulation in a water-soluble, solid matrix made from flash-flowable materials such as sweetening agents and the like. The oil is present in the matrix in finely divided micronized droplets which are dispersed in and entrapped by the surrounding matrix. The encapsulated liquid particles result from the flash-flow process, e.g., by melt-spinning a mixture of a water-soluble matrix with the oil to produce flakes or floss particles which can be used "as is" or subsequently further divided into a fine powder. The powder is formed of particles which in turn constitutes a matrix of the water-soluble protective coating and a micronized oil dispersion contained therein.

The preferred matrix materials are sweetening agents which are readily processed through flash-flow processing techniques. In one preferred embodiment the delivery system employs flavor oils which are intended to be used in comestible products. The combination of a sweet tasting water-soluble matrix and flavor or aromatic oil dispersed throughout provides a synergistic effect due to

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the concurrent release of both constituents. It is believed that the immediate delivery of the oil in the mouth provides a flavor/sweetener impact which is not only more intense but more true to the original flavor oil taste. This effect is due to the fact that more flavor components have been retained during processing and give a higher perception of intensity due to their intimate contact with the flash-flowable sweetener.

The flavor oils may be chosen from a host of materials which are suitable for the chosen application. Peppermint oil, for example, is one such oil which is commonly used in chewing gum and confectionery products and which when incorporated into the present delivery system provides a means of protecting the oil from loss of volatiles and degradation, yet produces immediate, up-front flavor release when placed in the mouth or other aqueous medium.

In one aspect of the invention, a particulate delivery system having a hydrophilic liquid dispersion or core and a solid water-soluble matrix is produced by means of flash-flow processing. The delivery system can be used in a variety of products for delivering hydrophobic oils, and in particular, flavor, fragrance and other aromatic oils. The delivery system is especially useful for instant and immediate delivery of the core material upon contact with moisture, e.g., when placed in the mouth. One particular application involves the use of the delivery system as dusting powders for chewing gum, confectionery products and baked goods.

The delivery system of the present invention provides uniform distribution of the hydrophobic liquid composition and retains the volatile and unstable components of the liquids which contribute to the overall character of the liquid. In the case of flavor oils and various aromatic



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oils, essential volatile components such as flavor notes and aromas are retained during processing and captured in the final product, which results in a more flavorful or fragrant product with high authenticity and trueness to its intended taste and/or smell.

The flash-flow process is essentially instantaneous and vaporization and/or degradation of the hydrophobic liquid composition, including other components such as sweeteners or fibers carried therewith, is substantially, if not completely reduced when compared to conventional spray-drying processing and simple mixing.

The delivery system can be used in applications such as flavor and sweetener dusting powders without the use of anti-caking materials such as corn starch. The delivery system emerges from the flash-flow process in a number of different forms, such as floss or flakes, but can be ground, pulverized or sieved into a fine particulate or powder without substantial loss of hydrophobic liquid or other components contained in the matrix. Cryogrinding using nitrogen is the preferred method of cominution. The retention of oil components in the inventive process is largely due to the fact that the liquid is in micronized form within the matrix rather than being simply adsorbed onto a carrier or enrobed within a coating.

The delivery systems also have a more uniform distribution of the hydrophobic liquid composition than can be achieved by simple mixing or spray drying techniques. One particular advantage is that a variety of flash-flowable materials can be used in conjunction with the hydrophobic oil to satisfy a host of applications. For example, spray-dried mixtures of cyclodextrins and flavors can be combined with the flash-flowable matrix materials to provide enhanced flavor-delivery systems.

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The flash-flow-formed delivery system retains substantially all of its flavor components and can be cryoground to a uniform particle size distribution. Cryogrinding has the unique advantage over conventional grinding of reducing heat build-up and subsequent loss of volatiles. This allows for a greater uniformity in particle size and shape than ordinary grinding, where frictional heat build-up can be problematic. Very fine powders can be produced using flash-flow processing followed by cryogrinding. These powders can be directly applied to comestibles and have particular application as flavor/sweetener delivery systems useful in chewing gum compositions or dusting powders on gum and confectionery surfaces. These powders readily adhere to chewing gum surfaces during rolling and scoring of the gum due to the uniform particle size and shape.

For a better understanding of the present invention, references made to the following description and its scope will be pointed out in the appended claims.

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#### BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a photomicrograph (500x) showing micronized peppermint oil in a flash-flow-formed matrix of Maltrin-365.

25 Figure 2 is a photomicrograph (500x) showing micronized peppermint oil in a flash-flow-formed particulate matrix of Maltrin-365 which has undergone cryogrinding.

30 Figure 3 is a photomicrograph (1250x) showing micronized peppermint oil in a flash-flow-formed matrix of an isomalt.

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Figure 4 is a photomicrograph (1250x) showing micronized peppermint oil in a flash-flow-formed particulate matrix of an isomalt which has undergone cryogrinding.

5        Figure 5 is a graph showing a comparison of volatilization retention of the flash-flow formed delivery system, with and without cryogrinding, as compared to simple mixture encapsulation under controlled temperature conditions.

10        DETAILED DESCRIPTION OF THE INVENTION

The present invention concerns the formation of a new solid delivery system for rapid release of hydrophobic liquids. The delivery system includes a matrix of water-soluble flash-flowable material having a micronized  
15        dispersion of hydrophobic liquid contained therein. The matrix is designed to be protective of the oil during processing, with little or no substantial loss of oil components during the delivery system formation, yet be readily releasable of the oil components in conditions of  
20        moisture. In particular, the oils are immediately released in aqueous medium and especially when placed in contact with the oral cavity or other moist conditions in or on the body where rapid release of oil is desirable.

The hydrophobic liquid can be chosen from a variety  
25        of materials such as oleaginous liquids, flavor or aromatic oils as well as mineral oil, glycerin, polyethylene glycol, and the like. Examples of oleaginous liquids include, without limitation, vegetable oils, fish oils, lard, lanolin, cocoa butter and mixtures thereof.  
30        It will be appreciated that those hydrophobic materials which are solid at room temperature can be used provided they are rendered sufficiently liquid to be dispersed within the matrix during processing. Alternatively, in

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cases where the oleaginous material can be rendered to a dispersible state with pre-heating without destroying or losing desired volatile components, such limited pre-heating may be employed. For example, animal fats such as tallow, lard or hydrogenated animal and/or vegetable oils can be employed in the present invention.

Hydrogenation or partially hydrogenated vegetable oils useful in the present delivery systems include such materials as corn oil, canola oil, cottonseed oil, sesame oil, soybean oil, grape-seed oil, sunflower oil, safflower oil, rapeseed oil, olive oil, peanut oil and the like. These oils, as well as the animal fat oils are ingestible and are therefore most commonly used in comestibles.

Other hydrophobic oils include those referred to as flavor oils or essences. These oils are generally derived from plant extracts, although may alternatively be synthetically derived. Peppermint oil, spearmint oil, cinnamon oil, oil of wintergreen, citrus oils and other fruit essences are the most commonly used flavor oils which are employed in the present invention. Flavor oils such as peppermint oil, spearmint oil and cinnamon oil are particularly harsh and create a burning sensation in the mouth if ingested in too high a quantity. The present invention allows for the use of smaller quantities than in typically comestible applications if desired due to the synergy which is achieved with the sweetener matrix. The micronized dispersion gives the perception that a greater quantity of flavor is present than the actual amount, thereby enhancing both the organoleptic impact with less flavor oil and eliminating the need for higher amounts. This is particularly useful in applications such as chewing gum compositions, where the addition of flavor oil at high concentrations to achieve a more intense flavor impact results in plasticization of the gum base components and sloppy chew characteristics.

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Examples of citrus or first oils and/or essences which are useful include a host of materials such as apple, apricot, banana, blueberry, cherry, grape, grapefruit, lemon, lime, orange, pear, peaches, pineapple, plum, raspberry, strawberry and the like. Mixtures and derivatives of these oils are contemplated.

Additional flavoring agents may be chosen from synthetic flavor oils and flavoring aromatics, and/or oils, oleo resins and extracts derived from plants, leaves, flowers, fruits and so forth, and combination thereof. For example, clove oil, bay oil, anise oil, eucalyptus oil, thyme oil, cedar leaf oil, oil of nutmeg, oil of sage, oil of bitter almonds and cassia oil may be used. Commonly used flavors include menthol, artificial vanilla, cinnamon derivatives, and various fruit flavors, whether employed individually or in admixture.

Flavorings such as aldehydes and esters including cinnamyl acetate, cinnamaldehyde, citral diethylacetal, dihydrocarvyl acetate, eugenyl formate, p-methylamisol, and so forth may also be used. Generally any flavoring or food additive such as those described in "Chemicals Used in Food Processing," pub 1274 by the National Academy of Sciences, pages 63-258 may be used.

Further examples of aldehyde flavorings include, but are not limited to acetaldehyde (apple); benzaldehyde (cherry, almond); anisic aldehyde (licorice, anise); cinnamic aldehyde (cinnamon); citral, i.e., alpha citral (lemon, lime); neral, i.e. beta citral (lemon, lime); decanal (orange, lemon); ethyl vanillin (vanilla, cream); hellotropine, i.e., piperonal (vanilla, cream); vanillin (vanilla, cream); alpha-amyl cinnamaldehyde (spicy fruity flavors); butyraldehyde (butter, cheese); valcraldehyde (butter, cheese); citronellal; decanal (citrus fruits); aldehyde C-8 (citrus fruits); aldehyde C-9 (citrus

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fruits); aldehyde C-12(citrus fruits); 2-ethylbutyraldehyde (berry fruits); hexenal, i.e., trans-2 (berry fruits); tolyl aldehyde (cherry, almond), veratraldehyde (vanilla); 2,6-dimethyl-5-heptenal, i.e. Melonal (melon); 2,6-dimethyl-5-heptenal, i.e., Melanal (melon); 2,6-dimethyloctanal (green fruit); and 2-dodecenal (citrus, mandarin); cherry; grape, strawberry shortcake; mixtures thereof; and the like.

Other specific flavor compounds such as ethylacetate, thiophene, ethylpropionate, ethyl butyrate, 2-hexanoate, 2-methylpyrazine, heptaldehyde, 2-octanone, limonene, and eugenol are also useful.

The hydrophobic oil content of the present delivery systems is generally in the range of about 0.02% to about 40% by weight of the delivery system. However, deviations from this range are certainly possible provided that the micronized dispersion of the oil in the matrix results as a result of the flash-flow process. Preferably, the oils are present in amounts of about 0.5% to about 20% by weight of the delivery system and most preferably about 2% to about 12%.

The matrix materials can be selected from any material which is capable of undergoing those physical and/or chemical changes associated with flash-flow processing. Flash-flow processing has been described in a number of commonly owned patents such as U.S. Patent 4,855,326, 5,001,532 and 5,096,492 from which the present application relates back, as well as 5,011,522 and 4,873,085, all of which are incorporated by reference herein.

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Those materials useful as matrices may be chosen from such classes as sugars or sugar derivatives. These types of materials are preferred because of their flash-flow capabilities and their wide variety of applications. The term sugar is meant to include those carbohydrates having a high glucose profile. A high glucose profile means that the carbohydrate has a large number of six-carbon mono and disaccharides as well as other glucose-based oligomers. Mono-, di-, tri- and polysaccharides and their derivatives may be employed. Examples include glucose, sucrose, maltose, lactose, arabinose, xylose, ribose, fructose, mannose, pentose, galactose, sorbose, dextrose, sorbitol, xylitol, mannitol, pentatol, maltitol, isomalt, sucralose, maltodextrin, polydextrose and mixtures thereof.

Other matrix materials include cellulosics and starches and their chemical and biological derivatives. Cellulosics, however, are generally added in combination with mono- and disaccharide-based materials because the cellulosics are not as easily processed alone using flash-flow techniques.

The delivery systems of the present invention have a substantially amorphous flash-flow-formed matrix. The terms "flash-flow" refers to a process of subjecting the feedstock, e.g. matrix material and hydrophobic oil, simultaneously to flash heating and applied physical force such that the solid matrix material experiences sufficient internal flow to transform it to a physically and/or chemically altered structure from that of the feedstock.

Flash-flow processing can be accomplished several ways. Flash heat and flash shear are two such processes which can be used. In the flash heat process, the feedstock material is heated sufficiently to create an internal flow condition which permits part of the feedstock to move at a subparticle level with respect to

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the rest of the mass and exit openings provided in the perimeter of the spinning head. The centrifugal force created in the spinning head flings the flowing feedstock material outwardly from the head so that it reforms with a changed structure. The force required to separate and discharge flowable feedstock is only centrifugal force which results from the spinning head. The flash heat process is one process for producing an amorphous matrix such as the sugar floss used in this invention.

10 In the flash shear process, a shearform matrix is formed by raising the temperature of the feedstock material which includes a non-solubilized carrier to a point where the carrier such as a saccharide-based material undergoes internal flow upon application of a fluid shear force. The feedstock is advanced and ejected while in internal flow condition, and subjected to disruptive fluid shear forces to form multiple parts or masses which have morphology different from that of the original feedstock.

20 The multiple masses are cooled substantially immediately after contact with the fluid shear force and are permitted to continue in a free-flow condition until solidified.

25 The feedstock material which can be used in a flash shear process includes but is not limited to a carrier such as a saccharide-based material. Other materials such as oleaginous materials can also be included in the feedstock.

30 It is important that the feedstock selected for a flash shear process have the ability to be processed without reliance upon dissolution. In the case of a saccharide based material, the feedstock is primarily a



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solid material which is subjected to the flash shear process.

5 The flash shear process can be carried out in an apparatus which has means for increasing the temperature of a non-solubilized feedstock and means for simultaneously advancing it for ejection. A multiple heating zone twin screw extruder can be used for increasing the temperature and advancing feedstock. The second element of the apparatus is a means for ejecting the feedstock in a condition for shearing it to provide the shearform matrix. The means for ejecting is in fluid communication with the means for increasing the temperature and is arranged at the point to receive the feedstock while it is in the internal flow conditions. 10 The means for ejecting the feedstock is preferably a nozzle which provides high pressure ejection of the feedstock material. For a description of various apparatus which can be used to produce the inventive delivery systems, see copending U.S. Serial No. (Docket 447-65), filed October 23, 1992 entitled "Process for Making Shearform Matrix", which is herein incorporated by reference. 20

A preferred flash-flow process used to form the inventive delivery systems involves spinning a feedstock in a "cotton candy" fabricating type machine. The spinning machine used to achieve a flash heat process can be a cotton candy type machine, such as the Econo-Floss Model 3017 manufactured by Gold Metal Products Company of Cincinnati, Ohio. It will be appreciated by those skilled in the art that any apparatus or physical process which provides similar forces and temperature gradient conditions can also be used. For simplicity in disclosing and describing this invention, the terms "flash heat" will be understood to mean a process which includes subjecting a feedstock to the combination of temperature, thermal 35

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gradients, flow, flow rates, and mechanical forces of the type produced in a cotton candy machine. The apparatus is operated at the temperature and speed which permit flash-flow of the feedstock without significant deterioration of ingredients including, for example, a crystallization control agent.

The matrix obtained in a melt-spinning process is in the form of a floss, fiber, particle, flake, spicule or any other generally non-descript amorphous aggregate.

Disclosures which relate to spinning substances with one or more sugars are found in commonly-assigned U.S. Patent Nos. 4,855,326, 4,873,085, 5,034,421, 4,997,856, 5,028,632, 5,034,421 and 5,096,492. These disclosures describe processing feedstock material by subjecting it to high speed spinning on a spinning head in which the substance is also subjected to heating against a heating element.

Additional additives can be added to the matrix/oil feedstock to achieve a variety of desired characteristics.

These include, without limitation, fillers, humectants, emulsifiers, surfactants, coloring agents, flavors, fragrances, sweetening agents, flash-flowable polymers, plasticizers and the like.

As previously mentioned, the inventive delivery system can be used to provide enhanced flavor and/or sweetness delivery due to the flavor oil being finely dispersed in the sweetener matrix. The quality of the flavor as well as the intensity is more predictably released into the oral cavity due to the unique physical characteristics which are created during the flash-flow process.

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By spinning the above-mentioned flavorant materials with the soluble melt spinnable carriers, even normally non-water soluble ingredients can be uniformly dispersed when contacted with water. The formation of the matrix is such that when added to a comestible such as chewing gum, for example, the flavor materials carried in the matrix rapidly dissolve when in contact with moisture. This unique property results in a dramatic flavor impact in the oral cavity.

10 In the case where two solid materials are to be used as the matrix, they may be combined prior to melt-spinning. For example, the matrix and the organoleptically perceivable material(s) may be combined by co-crystallization. Co-crystallization involves  
15 combining the ingredients in a heated state and thereafter allowing them to cool in a unified, crystallized manner. The unified structures are then reduced in size such as by being ground before being spun.

Other means of combining organoleptic perceivable materials with the matrix are also contemplated. For  
20 example, the matrix and flavorant may be combined in the spinning machine. In some cases, an oleaginous substance such as corn oil or polyvinylpyrrolidone (PVP), can be added to ensure uniform distribution of the flavor  
25 dispersion throughout the matrix of the spun product. For example, 2 parts oleaginous oil or a 2-3% solution of PVP may be added to the ingredients during the melt spinning.

The delivery system can also be compacted to less than 50% of the as-spun volume. Preferably, however, the  
30 delivery system is compacted to less than 30% and most preferably to less than 15% of the as-spun volume. As previously mentioned, the delivery system may also be reduced in particle size by milling before being added to comestibles.

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The delivery system of the present invention has been found to be especially effective as a dusting powder or confectionery coating for chewing gum, confectioneries tablets, nougats, dragies and the like.

5        In the particular application with respect to chewing gum compositions, the delivery system can be used as a dusting powder on the surface of the gum product. The inventive powders have distinct advantages over dusting  
10        powders conventionally used for a number of reasons. The physical form of the inventive delivery systems allows for immediate release of the flavor oil components when placed in contact with the mouth. This has particular  
15        significance in that up-front impact is achieved. Additionally, due to the unique formulation of the oil dispersion within the matrix, the intensity and quality of the flavor can be more easily controlled. Furthermore,  
20        the micronized flavor oil droplets within a sweetener matrix gives the perception a fuller flavor with less actual flavor being present. Since the delivery system was formed using flash-flow processing, more flavor oil  
25        components remain than with conventional simple mixing or other encapsulation techniques. The sweetening agents used as the matrix serve as anti-sticking or anti-blocking agents during the gum making process, i.e., particularly in the rolling and scoring process.

      The delivery system can be incorporated in conventional chewing gum compositions. These compositions typically contain a sweetener, a gum base and a flavor. The sweetener generally also serves as a bulking agent in  
30        sugared chewing gum compositions. One advantage of employing the inventive delivery system in chewing gum compositions is that the flavor can be directly incorporated with the bulking agent rather than in a separate step. Additional sources of flavor and/or

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sweetener can of course be combined with the delivery system and incorporated in the chewing gum composition.

With regard to the chewing gum compositions, the amount of gum base employed will vary greatly depending on various factors such as the type of base, consistency desired and other components used to make the final product. In general, amounts of about 5% to about 85% by weight of the final chewing gum composition are acceptable, with amounts of about 15% to about 30% by weight being preferred. The gum base may be any water-insoluble gum base well known in the art. Illustrative examples of suitable polymers in gum bases include both natural and synthetic elastomers and rubbers. For example, those polymers which are suitable in gum bases include, without limitation, substances of vegetable origin such as chicle, jelutong, gutta percha and crown gum. Synthetic elastomers such as butadiene-styrene copolymers, isobutylene-isoprene copolymers, polyethylene, polyisobutylene, polyvinylacetate and mixtures thereof are particularly useful.

The gum base composition may contain elastomer solvents to aid in softening the rubber component. Such elastomer solvents may comprise methyl, glycerol or pentaerythritol esters of rosins or modified rosins, such as hydrogenated, dimerized or polymerized rosins or mixtures thereof. Examples of elastomer solvents suitable for use herein include the pentaerythritol ester of partially hydrogenated wood rosin, pentaerythritol ester of wood rosin, glycerol ester of wood rosin, glycerol ester of partially dimerized rosin, glycerol ester of polymerized rosin, glycerol ester of tall oil rosin, glycerol ester of wood rosin and partially hydrogenated wood rosin and partially hydrogenated methyl ester of rosin, such as polymers of alpha-pinene or beta-pinene; terpene resins including polyterpene and mixtures thereof.

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The solvent may be employed in an amount ranging from about 10% to about 75% and preferably about 45% to about 70% by weight of the gum base.

5 A variety of traditional ingredients such as plasticizers or softeners such as lanolin, stearic acid, sodium stearate, potassium stearate, glycerol triacetate, glycerin and the like, including, natural waxes, such as paraffin waxes and microcrystalline waxes may also be incorporated into the gum base to obtain a variety of  
10 desirable textures and consistency properties. In accordance with the invention, however, these ingredients may be reduced in amount or in some cases, may be eliminated entirely. When present, these individual additional materials are generally employed in amounts of  
15 up to about 15% by weight and preferably in amounts of from about 3% to about 10% by weight of the final gum base composition.

The chewing gum may additionally include the conventional additives of coloring agents such as titanium  
20 dioxide; emulsifiers such as lecithin and glycerol monostearate; additional fillers such as aluminum hydroxide, alumina, aluminum silicates; calcium carbonate, and talc and combinations thereof; and additional  
25 flavoring agents. These fillers may also be used in the gum base in various amounts. Preferably, the amount of fillers when used will vary from about 4% to about 35% by weight of the final chewing gum.

The amount of delivery system used in the chewing gum composition will largely be a matter of preference. It is  
30 contemplated that the delivery system will be included in amounts of from about 0.25% to about 40% by weight of the final gum composition, with amounts of from about 1% to about 30% being preferred, and amounts of from about 1% to about 20% being most preferred.

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In addition to the inventive delivery system, the chewing gum composition may also optionally include one or more additional ingredients such as conventional polysaccharide-based bulking agents including sugars or sugar alcohols, flavor delivery systems, spray-dried flavors, liquid flavors, natural and/or artificial sweeteners and the like.

An important feature of the chewing gum compositions prepared in accordance with the present invention is the ability of the delivery system to rapidly dissolve when in contact with the moisture present in the oral cavity. This feature significantly decreases the propensity of the flavor oil to become entrapped and solubilized within the insoluble chewing gum cud during mastication.

The chewing gum compositions of the present invention may be prepared by combining the water-insoluble gum base portion and the water-soluble flavor portion including the novel flavor/sweetener delivery system matrix according to conventional chewing gum processing techniques.

For illustrative purposes, a method of preparing the novel chewing gum compositions is as follows:

A suitable chewing gum base is first melted. Softeners and bulking agents such as sugar alcohols if desired may be added slowly with stirring thereafter. The inventive delivery system is then added and mixing is continued until a homogeneous mass is achieved. Optionally, additional flavor oils or spray dried flavors may be added as well. The mass may then be rolled, scored, dusted and wrapped in any manner known in the art.

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With regard to the preparation of other types of comestibles, the spun matrix may also be added in a conventional manner. For example, in the case of pressed tablets, the flavor/sweetener delivery system may be dry  
5 blended with the remaining tablet ingredients and the mixture thereafter compressed into final tablet form. In the case of dentifrices, denture creams and cleansers, the products also benefit from incorporation of the delivery system in their formulations. In short, the matrix may be  
10 added to various comestibles in a manner similar to that which the skilled artisan currently uses to add conventional water-soluble comestible ingredients.

The present invention also provides for enhanced delivery of hydrophobic materials and allows these  
15 materials which are normally difficult to disperse in water, readily dispersible when placed in aqueous solution.

The delivery systems of the present invention can include a number of additional components which can be  
20 dispersed along with the hydrophobic liquid. These components can be pre-mixed with the liquid and added to the feedstock mixture of matrix and oil, or added concurrently with the matrix material and oil. For example, various sweeteners, such as natural or synthetic  
25 sweeteners can be combined with flavor oils by direct addition or in spray-dried form. Additionally, the flavor oil may be adsorbed on or incorporated in a carrier material prior to admixture with the matrix materials. Such carrier materials may include other flash-flowable  
30 materials or may be materials which are not easily flash-flow processed alone but can be added in amounts up to 50% of the total composition and combined with other more flash-flowable materials.



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In one particular embodiment, micron-sized synthetic, amorphous silica has been used as a carrier for the hydrophobic liquids. These silicas have a unique combination of uniformity, chemical inertness, large surface area and porosity which make them highly adsorptive. These silicas can be manufactured with precisely controlled surface area, porosity and particle size distribution, which make them extremely useful in the inventive compositions. Commercially available silicas of this kind are sold under the trademarks SYLOID and SYLOX by W.R. Grace & Co., Baltimore, MD. These materials are specifically intended for use in dispersions and suspensions. Using these materials, flavor oils can be adsorbed onto their surfaces and into their pores and then added to the feedstock of matrix material to form the inventive delivery systems. In this manner, additional controlled release characteristics can be imparted to the delivery systems, as well as adding further stabilization and protective features to the oils against volatilization and oxidation. These silica compounds also have ionic and hydrogen bond affinity for certain flavor component chemical groups, which affinity serves to strengthen flavor retention and consequently allows for increased delayed release capabilities and stabilization characteristics.

Additional materials which can be as carriers for the flavor oils prior to incorporation with the inventive delivery system include maltodextrins, such as spray-dried maltodextrin marketed under the tradename M100 (10 DE) by Grain Processing Corporation, Muscatine, IA, as well as agglomerated maltodextrin (10 DE) sold under the tradename Micropor Buds 1015A, by E. Staley Manufacturing Co., Decatur, IL. These materials are also porous and allow for flavor retention. Polydextrose and microcrystalline cellulose are also useful in this regard, as are a number of other adsorbent materials.

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In one embodiment, the sweetener matrix or other matrix material can be combined with a cellulosic material such as microcrystalline cellulose and flash-flow processed. The resultant solid can then be further  
5 processed in accordance with present invention by adding artificial sweeteners and/or flavors and the like to it and again flash-flow processed.

Microcrystalline cellulose can also be used as the primary matrix material and combined with natural or  
10 artificial sweeteners, such as those enumerated herein, to form a delivery system which can be used "as is" or further combined with flavor oil and either flash-flow processed further or added directly to a comestible or other useful product, such as a pharmaceutical, cosmetic,  
15 dry food or drink mix, cereal, personal hygiene product or the like.

The present delivery systems are also useful in antacid compositions and especially those compositions designed in chewable dosage forms. For example, these  
20 compositions generally have sodium, calcium or magnesium carbonates present and in some cases aluminum hydroxide.

The processes of the present invention have also been shown to be especially useful in forming sweet and flavorful products which have less sugar present. For  
25 example, an artificial sweetener and/or flavor can be combined with corn syrup solids as the matrix and flash-flow processed with the inventive delivery system. The delivery system can then be diluted with non-sweet fillers such as starch or polydextrose and the like and added to  
30 the final product, e.g. a comestible, thereby reducing the amount of corn solids present in the final product.

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Invert sugar has also been found to be effective when used in the present delivery system, especially in combination with other flash-flavorable sweeteners because it produces a product having less hygroscopicity. Other materials which perform this function may also be used, as well as materials which hold water well, such as humectants, in applications where water content in the processing or final delivery system is to be controlled.

#### EXAMPLES

The following examples serve to provide further appreciation of the invention, but are not meant in any way to restrict the effective scope of the invention. Unless indicated otherwise, the Econo-Floss machine referred to above was used to form delivery systems.

##### EXAMPLE 1

Inventive delivery systems were prepared using sucrose as the matrix and spearmint flavor oil as the hydrophobic oil. In this example, 100 grams of sucrose were first hand mixed with 2 grams of spearmint flavor oil until a uniform mixture was obtained. The mixture was then flash-flow processed at a medium setting and approximately 3500 RPM. The resultant delivery system produced was a fine floss containing micronized droplets of flavor oil which was thereafter milled to a particle size of about 50 microns. The delivery system displayed rapid solubility and high flavor impact when placed in the mouth.

##### EXAMPLE 2

This example uses corn syrup solids as the matrix material, along with orange flavor oil and corn oil. In this example, 100 grams of corn syrup solids were hand

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mixed with 4 grams of orange flavor oil and 100 milligrams of sucralose. Thereafter, 2 grams of Mazola® corn oil was geometrically added to the mixture using a mortar and pestle. The mixture was then flash-flow processed at a low setting to produce the inventive delivery systems having a flake-like appearance and sweet, orangy taste.

### EXAMPLE 3

In this example, 100 grams of polydextrose K was hand mixed with 100 milligrams of the artificial high intensity sweetener sucralose until a uniform mixture was obtained. Thereafter, 2 grams of Mazola® corn oil was geometrically added to the mixture using a mortar and pestle. The resultant mixture was then flash-flow processed at a low setting to produce the inventive delivery system having a chip-like appearance and intense sweetness.

In Examples 4-6, the delivery systems prepared in Examples 1-3 are incorporated into chewing gum compositions in the amounts set forth below. The resultant chewing gum compositions display a rapid perception of flavor with up-front high impact.

### EXAMPLE 4

#### Chewing Gum

	<u>Ingredient</u>	<u>Percentage by Weight</u>
	Delivery System (Example 1)	13.00
25	Gum Base	33.00
	Carbohydrates	44.33
	Softeners	9.50
	Colorant	<u>0.17</u>
		100.00

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EXAMPLE 5Sugarless Chewing Gum

	<u>Ingredient</u>	<u>Percentage by Weight</u>
	Delivery System (Example 2)	9.00
5	Gum Base	30.00
	Carbohydrates	51.33
	Softeners	9.50
	Colorant	<u>0.17</u>
		100.00

10

EXAMPLE 6Sugarless Chewing Gum

	<u>Ingredient</u>	<u>Percentage by Weight</u>
	Delivery System (Example 3)	3.85
	Gum Base	23.00
15	Carbohydrates	63.33
	Softeners	9.50
	Colorant	0.17
	Flavor Oil	<u>0.15</u>
		100.00

20        In the following example, the delivery system of Example 1 was included in a pressed tablet to demonstrate that the soluble matrix also provides high flavor impact in comestibles other than chewing gums.

EXAMPLE 7

25

Pressed Tablets

	<u>Ingredient</u>	<u>Percentage by Weight</u>
	Delivery System (Example 1)	22.00
	Sugar	77.02
	Copper Gluconate	0.75
30	(Breath Freshener)	
	Lubricant	<u>0.23</u>
		100.00

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Examples 8 and 9 demonstrate the use of the novel delivery systems in oral hygiene products. The high flavor impact provides an added organoleptic experience to otherwise mundane daily routines.

5

EXAMPLE 8Tooth Powder

	<u>Ingredient</u>	<u>Percentage by Weight</u>
	Delivery System (Example 3)	2.40
	Silica Hydrogel	95.10
10	Zinc Chloride	0.50
	Sodium Fluoride	0.22
	Sodium Gluconate	0.28
	Sodium Methyl Cocoyltaurate	<u>1.50</u>
		100.00

15

EXAMPLE 9Dentifrice Composition

	<u>Ingredient</u>	<u>Percentage by Weight</u>
	Delivery System (Example 2)	4.30
	Glycerin	25.00
20	Silicone Dioxide	21.50
	HMP (Hexaphos)	6.00
	Silica	3.00
	Sodium Lauryl Sulfate	1.20
	Sodium Hydroxide (50% solution)	1.00
25	Xanthan Gum	1.00
	Sodium Benzoate	0.50
	Titanium Dioxide	0.50
	Fluoride	0.22
	Deionized Water Q.S.	100.00

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EXAMPLE 10

This example is intended to demonstrate the use of amorphous micron-sized silica as adsorbent carriers for flavor oils which are incorporated into the inventive delivery systems for the purpose of achieving delayed release. A mixture of peppermint oil (75 gr.) and amorphous silica (75 gr.) (SYLOID 244 FP, Davison Chemical) was mixed in a Hobart mixer. This mixture is then added to polydextrose (2,348 gr.) and mixing is continued. The mixture is then flash-flow processed on an Econo-Floss spinning machine at 3600 RPM at 190°C to yield a fine flake-like material. This delivery system is then incorporated in an amount of about 10% by weight into the chewing gum composition of Examples 4 and 5, in place of the delivery systems of Examples 1 and 2 respectively. The resultant chewing gum products demonstrated a delayed flavor release due to the affinity of the flavor for the silica and a reduced tendency of flavor oil to over-plasticize the gum base.

20

EXAMPLE 11

Examples 11 through 14 demonstrate the usefulness of the inventive delivery systems as dusting powders on chewing gum products, to aid in the rolling and scoring processes and prevent sticking and binding in the overall handling of the gum, both during processing and wrapping.

A flavor delivery system for delivery of up-front flavor in the form of a powder is prepared for use on the surface of a chewing product. 75 gr. of peppermint oil was mixed with 75 gr. of SYLOID 244FP (obtained from Davison Chemical) to which 1.5 gr. of aspartame and 0.5 gr. of saccharin were added followed by mixing. This mixture was then gradually added to 2,348 gr. of Palatinit® Type PF (an isomalt obtained from Süßungsmittel

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GmbH, Mannheim, Germany) in a Hobart mixer. The mixture was then flash-flow processed on an Econo-Floss spinning machine at 3600 RPM at 190°C yielding a white floss. The material was cryoground with liquid nitrogen to produce a fine white powder.

The powder was applied to the surface of a chewing gum mass which was then rolled between pressure rollers and formed into a slab. The powder adhering to the gum surface provided an immediate, pleasant sweet mint flavor when placed on the tongue.

#### EXAMPLE 12

The following flavor delivery system was prepared for use on the surface of a chewing gum product. 10.0 gr. of peppermint oil was mixed with 5.0 gr. of SYLOID 244FP to which 1.0 gr. of flour salt was added followed by mixing. This mixture was then gradually added to 394 gr. of Sucrose 10x in a Hobart mixer. The mixture was then flash-flow processed on an Econ-Floss spinning machine (which was modified to provide variable speed control and variable heat control) at 3,300 RPM at 190°C, yielding a white floss. The material was cryoground with liquid nitrogen to produce a fine white powder.

The powder was applied to the surface of a chewing gum mass which was then rolled between pressure rollers and formed into a slab. The powder adhering to the gum surface provided an immediate, pleasant sweet mint flavor when placed on the tongue.

#### EXAMPLE 13

The following delivery system was prepared for use on the surface of a chewing gum product. 1.6 gr. of peppermint oil was mixed with 2 gr. of flour salt to which



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2.0 gr. of aspartame was added followed by mixing. This mixture was then gradually added to 494.4 gr. of polydextrose in a Hobart mixer. The mixture was then flash-flow processed on an Econo-Floss spinning machine (which was modified to provide variable speed and variable heat) at 3300 RPM at 140°C yielding a white floss. The material was cryoground with liquid nitrogen to produce a fine white powder.

The powder was applied to the surface of a chewing gum mass which was then rolled between pressure rollers and formed into a slab. The powder adhering to the gum surface provided an immediate, pleasant sweet mint flavor when placed on the tongue.

#### EXAMPLE 14

The following flavor delivery system was prepared for use on the surface of a chewing gum product. 100 gr. of peppermint oil was mixed with 5 gr. of SYLOID 244FP (obtained from Davison Chemical) to which 1.0 gr. of flour salt was added followed by mixing. This mixture was then gradually added to 394 gr. of corn syrup solid PE 36 (obtained from Hubinger) in a Hobart mixer. The mixture was then flash-flow processed on an Econo-Floss spinning machine (which was modified to provide variable heat and variable speed) at 3300 RPM at 145°C yielding a white floss. The material was cryoground with liquid nitrogen to produce a fine white powder.

The powder was applied to the surface of a chewing gum mass which was then rolled between pressure rollers and formed into a chewing gum piece. The powder adhering to the gum surface provided an immediate, pleasant sweet mint flavor when placed on the tongue.

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The powders of Examples 1-4 can also be used to provide up-front flavors bursts on mints, candies, baked goods, dentifrices, pharmaceuticals and the like.

#### EXAMPLES 15 AND 16

5       The following delivery systems were made in accordance with the process of the present invention using a spinning head temperature of about 140°C for Example 15 and 190°C for Example 16. Examples demonstrate the physical form of the delivery systems to be a matrix  
10       having a finely divided or micronized dispersion of flavor droplets distributed therein.

##### Example 15

97% Maltrin - 365 (a maltodextrin DE 36)  
3% Peppermint Oil

##### 15       Example 16

97% Palatinit® (an isomalt)  
3% Peppermint Oil

20       The delivery system of Example 15 was then photographed using conventional phase-contrast microscopy techniques to clearly discern the oil phase from the amorphous solid matrix. Figures 1 and 2 relate to Example 15, whereas Figures 3 and 4 relate to Example 16.

25       Figure 1 is a photomicrograph at 500x magnification. The peppermint oil dispersion is clearly evident as those areas where birefringence (double circle areas) is present. The liquid can actually be seen to move within the encapsulated packet of the matrix and is easily discernable from air pockets, which lack both birefringence and movement.

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Figure 2 shows the delivery system of Example 15 at 1250x magnification subsequent to further cominution into a powder by cryogrinding with nitrogen. The minute flavor droplets are still present in the resultant powder, as  
5 seen in the birefringent areas of the photomicrograph, as well as through movement of the oil as seen under the microscope.

Figure 3 is a photomicrograph (500x) of the delivery system of Example 16. Again the flavor dispersion is  
10 evident from the picture.

Figure 4 shows the same delivery system subsequent to cryogrinding with nitrogen to a fine powder. The same physical results are present, demonstrating that the uniquely fine dispersion formed within the matrix remains  
15 even after grinding.

#### EXAMPLES 17-19

These examples are intended to demonstrate the ability of the inventive process and delivery system made therefrom to better protect the volatile flavor  
20 components.

A simple mixture of 92% Maltrin-365 (DE-36) and 8% peppermint oil was prepared in a Hobart mixer. This mixture was labelled Example 17. A sample of this mixture was used as the feedstock for preparing a delivery system  
25 of the present invention using an Econo-Floss machine (140°F, 3600 RPM). This delivery system was labelled Example 18.

Finally, a sample of Example 18 was cryoground using nitrogen and labelled Example 19.

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Each of Examples 17, 18 and 19 were incubated in a temperature controlled environment (40°C) for an extended period of time for the purpose of measuring volatile component loss, or in other words, determining relative flavor retention. Figure 5 graphically depicts the percent oil remaining at various intervals of time. It is abundantly clear from the graph that the inventive delivery systems of the present invention (Examples 18 and 19) have retained the flavor over 168 hours at 40°C significantly better than the simple mixture of Example 17. In fact, Example 18 shows only a nominal loss of flavors as compared to the simple mixture of Example 17. This is believed to be due to the unique physical properties resulting from the novel form of the delivery system.

While there have been described what are presently believed to be the preferred embodiments of the present invention, those skilled in the art will realize that changes and modifications may be made thereto without departing from the spirit of the invention, and it is intended to claim all such changes and modifications as fall within the true scope of the invention.

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WHAT IS CLAIMED IS:

1. A solid delivery system for rapid release of hydrophobic liquids comprising a water-soluble flash-flow formed matrix containing a micronized dispersion of a substantially hydrophobic liquid.
2. The delivery system of claim 1 wherein the hydrophobic liquid is an oleaginous material.
3. The delivery system of claim 1 wherein the hydrophobic liquid is a flavor or fragrance oil.
4. The delivery system of claim 2 or 3 wherein the matrix is a sugar or sugar derivative.
5. The delivery system of claim 4 wherein the matrix is selected from the group consisting of sucrose, maltose, dextrose, ribose, fructose, lactose, glucose, arabinose, mannose, pentose, sorbose, xylose, galactose, sorbitol, mannitol, galactitol, lactitol, maltitol, maltooligosaccharides, pentatol, isomalt, xylitol, sucralose, maltodextrin, polydextrose and derivatives and mixtures thereof.
6. The delivery system of claim 5 wherein the flavor oil is selected from the group consisting of natural and artificial flavors.
7. The delivery system of claim 6 wherein the flavor oil is a natural or synthetic plant oil or essence.
8. The delivery system of claim 7 wherein the flavor is selected from the group consisting of peppermint oil, spearmint oil, cinnamon oil, oil of wintergreen, nut oil, licorice, vanilla, citrus oils, fruit essences and mixtures thereof.

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9. The delivery system of claim 8 wherein the citrus oil and fruit essences are selected from apple, apricot, banana, blueberry, cherry, coconut, grape, grapefruit, lemon, lime, orange, pear, peaches, pineapple, plum, raspberry, strawberry and mixtures thereof.

10. The delivery system of claim 4 wherein the oleaginous material is selected from the group consisting of a vegetable oil, mineral oil, or animal fat.

11. The delivery system of claim 10 wherein the oleaginous material is an edible oil.

12. The delivery system of claim 1 wherein the flash-flow-formed matrix is formed under conditions of flash heat, or flash shear.

13. The delivery system of claim 1 wherein the hydrophobic liquid is present in the matrix in a range of about 0.25% to about 40% by weight.

14. The delivery system of claim 5 wherein there is additionally incorporated a sweetener selected from the group consisting of dipeptide and amino acid-based sweeteners, saccharin salts and its free acid, cyclamate salts, acesulfame and its salts, hydrogenated starch hydrolysate, talin, thaumatin, steroside, dihydrochalcone and mixtures thereof.

15. A method of producing particulates useful for delivering hydrophobic liquids, said particulates having dispersed therein a micronized hydrophobic liquid, comprising the step of subjecting a flash-flowable material to flash-flow conditions to form a solid matrix of the flash-flowable material containing a micronized dispersion of the hydrophobic liquid.

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16. The method of claim 15 wherein the solid matrix is reduced to finer particles by grinding, pulverizing or sieving.

17. The method of claim 16 wherein the solid matrix is reduced to a finer particles by cryogrinding.

18. The method of claim 15 wherein the flash-flowable material is a saccharide-based compound.

19. The method of claim 18 wherein the flash-flowable material is selected from the group consisting of sucrose, maltose, dextrose, ribose, fructose, lactose, glucose, arabinose, mannose, pentose, xylose, galactose, sorbitol, mannitol, galactitol, lactitol, maltitol, maltooligosaccharides, pentatol, isomalt, xylitol, sucralose, maltodextrin, polydextrose and derivatives and mixtures thereof.

20. The method of claim 15 wherein the flash-flowable matrix additionally incorporates a flash-flowable non-saccharide based polymer.

21. The method of claim 15 wherein the non-saccharide based polymer is a thermoplastic or cellulosic.

22. The method of claim 15 wherein the hydrophobic liquid is a flavor or fragrance oil.

23. The method of claim 15 wherein the flash-flowable material and/or the hydrophobic oil is pre-mixed with a material selected from the group consisting of fillers, sweeteners, coloring agents, humectants, plasticizers, emulsifiers and mixtures thereof.

24. The method of claim 23 wherein the pre-mix comprises a flavor oil and a sweetener.

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25. The method of claim 15 wherein the flavor oil is absorbed onto a carrier material prior to flash-flow processing.

26. The method of claim 15 wherein the carrier material is a synthetic amorphous silica or porous maltodextrin aggregate.

27. A chewing gum composition comprising:

a) a gum base; and

b) a solid sweetener and flavor delivery system comprising a water-soluble flash-flow-formed matrix  
5 containing a micronized dispersion of flavor oil.

28. The chewing gum composition of claim 27 wherein the water-soluble flash-flow-formed matrix is a saccharide-based material.

29. The chewing gum composition of claim 27 wherein the flavor oil is pre-mixed with a synthetic amorphous silica prior to incorporation in the matrix.

30. A chewing gum product having a core portion comprising gum base, sweetener and flavor and having deposited on the surface of said core portion a powdered rolling material comprising particles of flash-flow-formed  
5 saccharide-based material containing a micronized dispersion of a flavor oil.

31. The chewing gum product of claim 30 wherein the saccharide-based material is selected from the group consisting of sucrose, maltose, dextrose, ribose, fructose, lactose, glucose, arabinose, mannose, pentose,  
5 xylose, galactose, sorbitol, mannitol, galactitol, lactitol, maltitol, maltooligosaccharides, pentatol, isomalt, xylitol, sucralose, maltodextrin, polydextrose and derivatives and mixtures thereof.



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32. The chewing gum product of claim 31 wherein the saccharide-based material contains an artificial sweetener.

33. A comestible product having enhanced flavor and sweetness comprising an edible flash-flow-formed delivery system having a hydrophobic micronized dispersion contained in a flash-flowable material.

34. The comestible product of claim 33 wherein the flash-flowable material is a saccharide-based material.

35. A flash-flow-formed sweetener delivery system comprising a solid amorphous saccharide-based matrix material and an artificial sweetener dispersed therein.

36. A delivery system for flavors and sweeteners comprising a flash-flowable saccharide-based matrix containing a sweetener or flavor dispersed therein formed by flash-flow processing.

37. The delivery system of claim 1 incorporated into a cosmetic composition.

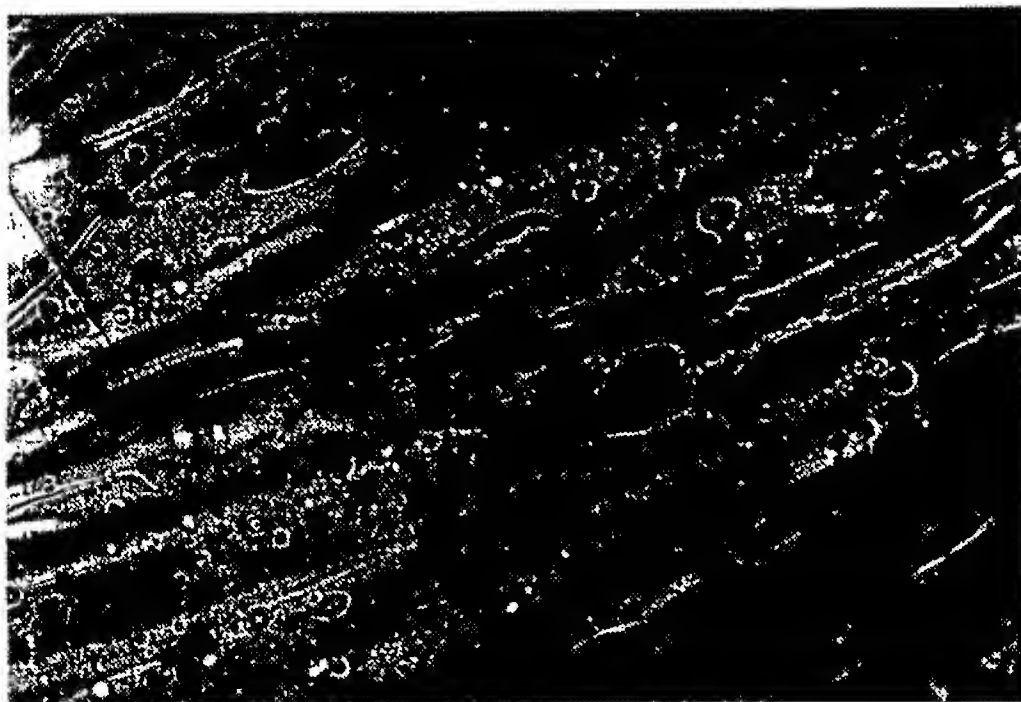
38. The delivery system of claim 1 incorporated into a dentifrice composition.

39. The delivery system of claim 4 incorporated into a comestible.

40. The delivery system of claim 4 incorporated into a confectionery product.

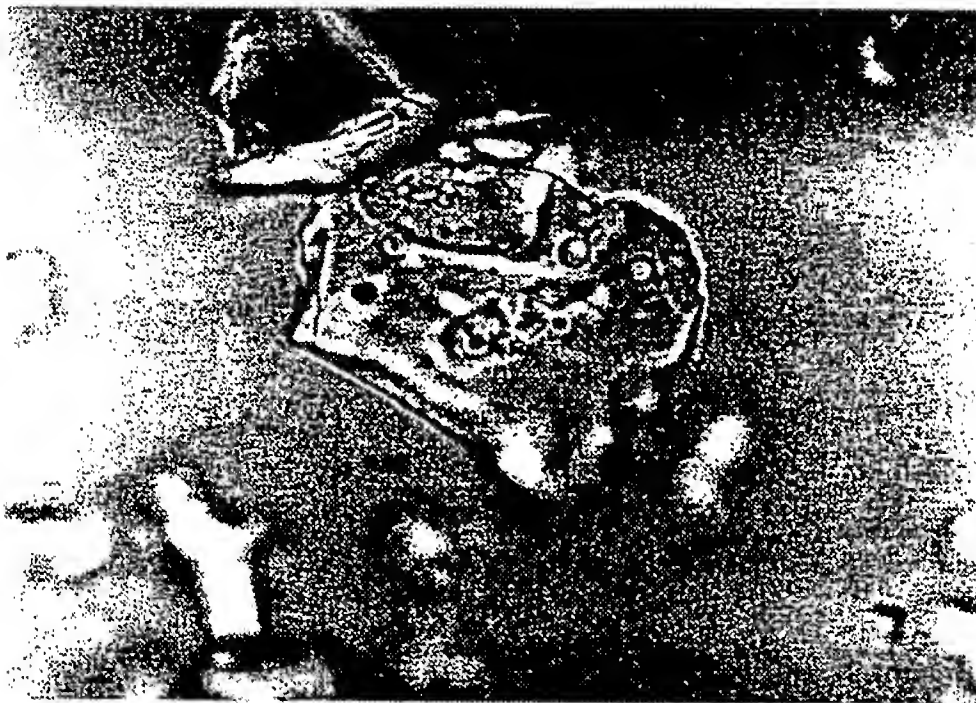
41. The delivery system of claim 4 incorporated into a baked product.

Fig. 1



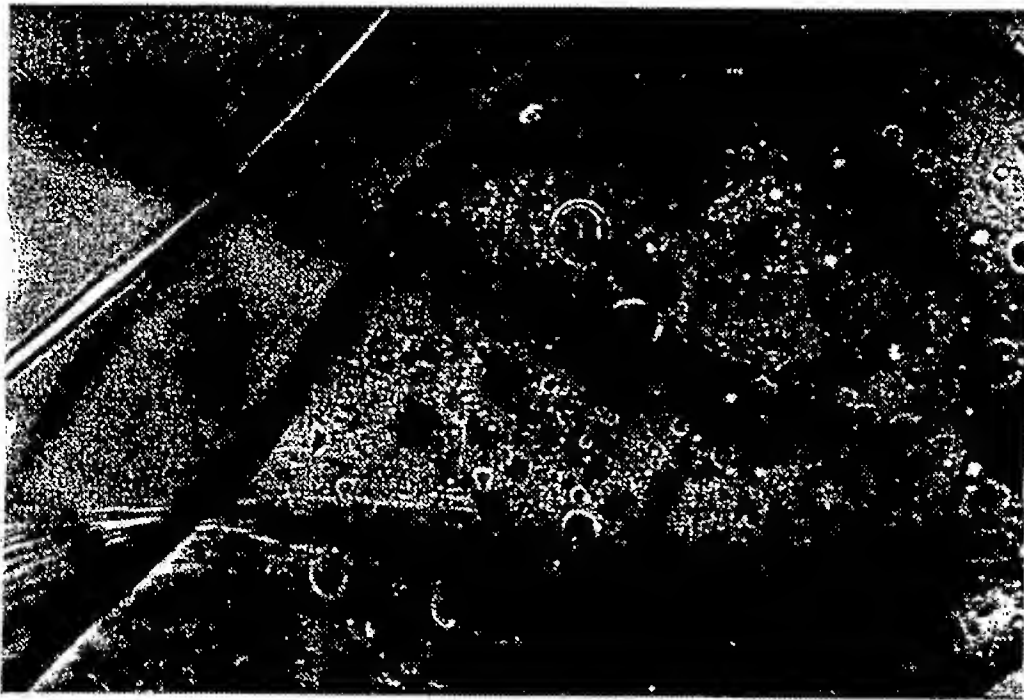
3% PEPPERMINT OIL IN  
97% MALTRIN-365  
500X

Fig. 2



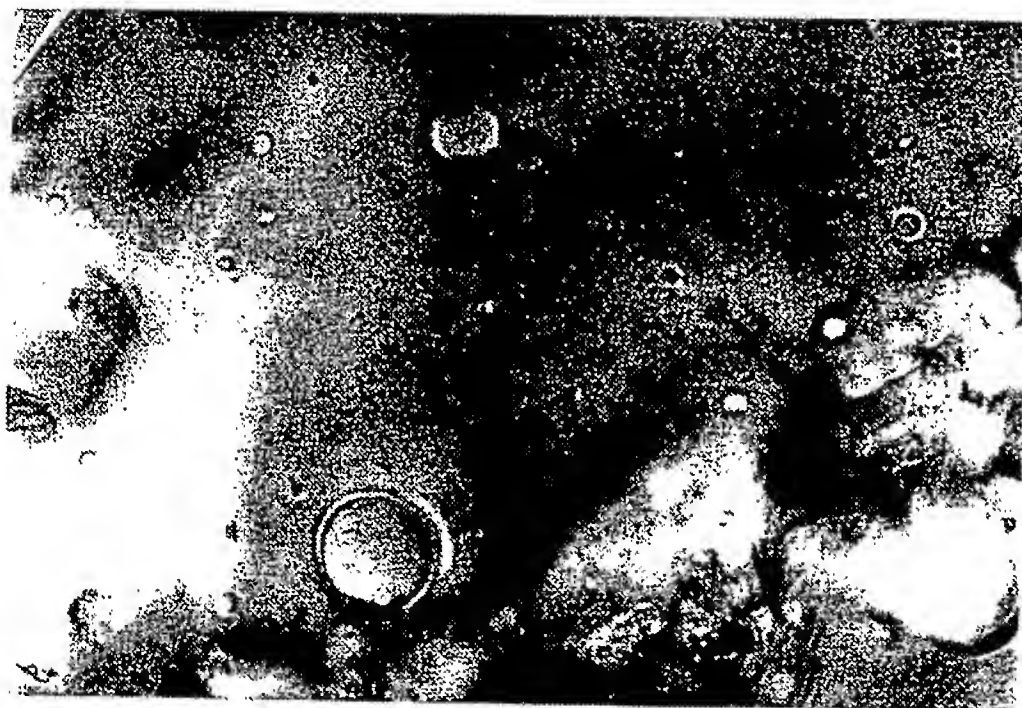
3% PEPPERMINT OIL IN  
97% MALTRIN-365 (CRYOGROUND)  
1250X

Fig. 3



3% PEPPERMINT OIL IN  
97% PALATINIT PF  
500X

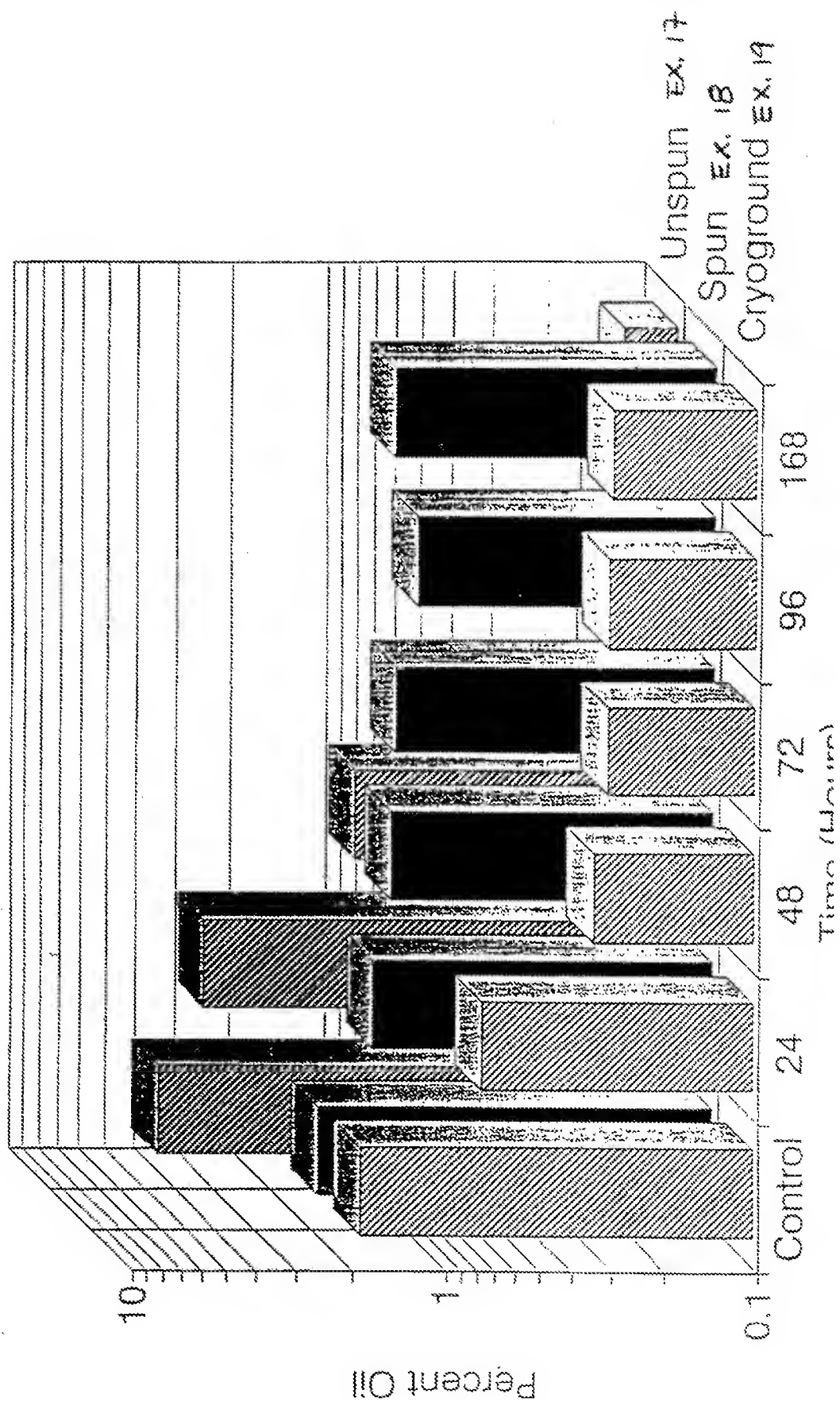
Fig 4



3% PEPPERMINT OIL IN  
97% PALATINIT (CRYOGROUND)  
1250X

Fig. 5

Incubated at 40°C



## INTERNATIONAL SEARCH REPORT

PCT/US92/09447

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : A23G 3/30; A23L 1/222; A23P 1/04

US CL : 426/3, 4, 5, 6, 96, 98, 650, 651

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 426/3, 4, 5, 6, 96, 98, 650, 651

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 3,930,043 (WARNING ET AL.) 30 December 1975. See the entire document.	1-41
Y	US, A, 3,723,134 (CHIVERS) 27 March 1973. See the entire document.	1-41
Y	US, A, 3,595,675 (ASH ET AL.) 27 July 1971. See the entire document.	1-41
Y	US, A, 4,496,592 (KUWAHARA ET AL.) 29 January 1985. See the entire document.	1-41
Y	US, A, 4,855,326 (FUISZ) 08 August 1989. See the entire document.	1-41

☐ Further documents are listed in the continuation of Box C.☐ See patent family annex.

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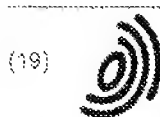
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Authorized officer *my mess*  
JEANETTE M. HUNTER

Facsimile No. NOT APPLICABLE

Telephone No. (703) 308-3849



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European Patent Office  
Office européen des brevets



(11) EP 1 197 153 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:  
17.04.2002 Bulletin 2002/16

(51) Int. Cl. 7: **A23D 7/00**, A23D 7/015,  
A23L 1/24, A23L 1/307,  
A23L 2/52

(21) Application number: **02075153.3**

(22) Date of filing: **29.07.1998**

(84) Designated Contracting States:  
**BE DE DK ES FI FR GB IE NL SE**

(30) Priority: **22.08.1997 US 916877**

(62) Document number(s) of the earlier application(s) in  
accordance with Art. 76 EPC:  
**98202536.3 / 0 897 671**

(71) Applicant:  
• **UNILEVER N.V.**  
**3013 AL Rotterdam (NL)**  
Designated Contracting States:  
**BE DE DK ES FI FR NL SE**  
• **UNILEVER PLC**  
**London EC4P 4BQ (GB)**  
Designated Contracting States:  
**GB IE**

(72) Inventors:  
• **Patrick, Matthew**  
**Englewood Cliffs, New Jersey 07632 (US)**  
• **Traska, Elexander Włodzimierz**  
**Englewood Cliffs, New Jersey 07632 (US)**

(74) Representative: **Van Velzen, Maaike Mathilde et al**  
**Unilever N.V.**  
**Patent Department**  
**Postbus 137**  
**3130 AC Vlaardingen (NL)**

Remarks:

This application was filed on 14 - 01 - 2002 as a  
divisional application to the application mentioned  
under INID code 62.

(54) **Aqueous dispersions or suspensions**

(57) Aqueous dispersions of plant sterols and other  
high melting lipids. The dispersions are useful in  
spreads and other food products. The dispersions pro-  
vide structure to the food products and their use can per-  
mit minimization or elimination of saturated fats and

trans fatty acids. The invention is also directed to a pro-  
cess for making the dispersions and to water and fat con-  
tinuous spreads and other food products including the  
high melting lipids.

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## Description

Background of the invention

5 [0001] Hydrogenation, fractionation and interesterification are the most common processes utilized within the fats and oils industry to modify the chemical and/or physical properties of conventional triglyceride fats or other lipids to improve their utility and functionality, e.g. to modify melting points, increase hardness or the solid fat content, increase oxidative stability, etc. These processes may be used individually or can be combined to produce fats and oils with very specific characteristics. When employing these processes to increase hardness or the solid fat content in the simplest of terms, these processes rely on the reduction of unsaturation or the increase of saturation and/or the formation of trans fatty acids.

10 [0002] Considerable attention has been drawn in recent years to the relatively high total fat, saturated fat and trans fat content of the typical diet.

15 [0003] Related health issues which have been greatly publicized are elevated cholesterol levels and low HDL/LDL cholesterol ratios reportedly resulting from ingestion of saturated fats and cholesterol. And, in addition to saturated fats, some reports have implicated trans fatty acids, which are generated when liquid oils are partially hydrogenated to increase their solid fat content.

20 [0004] Phytosterols are sterols found in plants. While these compounds have long been touted for their cholesterol lowering effects, they tend to be very high melting compounds (melting points around 150°C), and they are difficult to formulate into consumer food products due to poor solubility in fats and immiscibility in water. This solubility problem has been partially mitigated by esterification of the sterol. Nevertheless, this still limits use of sterols to food compositions with moderate to high fat contents.

[0005] U.S. Patent No. 5,156,886 discloses sterols used in chewing gums. An emulsifier is added to decrease viscosity.

25 [0006] U.S. Patent No. 5,445,811 is directed to phytosterols used in an oil-in-water emulsion intended for intravenous administration as a contrasting agent for visualizing the presence of tumors. Synthetic emulsifiers of the type normally used for preparing oil-in-water emulsions are said to be of particular interest. The weight ratio between emulsifier and cholesterol or phytosterol can range between 1:1 and 1:2. In Example 2, 0.06g cholesterol is used per 100 ml of mixture. The mixture is homogenized and autoclaved at 121°C. In Example 1, 2g of cholesterol are used and the mean particle size of the emulsion is 0.25  $\mu$ m. In Example 6, 1.6g of cholesterol per 100ml mixture are used. It is said that the mean size of the emulsion particles will preferably be beneath 1  $\mu$ m.

30 [0007] U.S. Patent No. 5,244,887 is directed to the use of plant stanols to reduce cholesterol absorption from foods. It is said that the greatest effectiveness is obtained when the stanols are evenly distributed in finely divided form throughout the food product or beverage. This can be accomplished by dissolving the stanols in a solubilizing agent such as vegetable oil, monoglyceride, diglycerides, tocopherols, and mixtures thereof and making suspensions or emulsions of the solubilized stanols in carriers such as water, alcohol, polyols and other edible compounds or by suspension of the stanols in an emulsion. Solubilizing agents such as monoglycerides and diglycerides are mentioned. A preferred food additive is said to comprise, in addition to 25% stanols, 74.8% vegetable oil and tocopherol. The compounds of the invention are used as food additives to foods such as meats, eggs, and dairy products. The stanols are said to remain in solution or uniformly suspended.

35 [0008] U.S. Patent No. 4,160,850 mentions low fat margarine products having less than 60 wt. % fat. The invention is directed to a shelf stable mix suitable for consumer preparation of a spreadable butter substitute having from about 20-80% oil and from about 10 to about 80% water. A water-in-oil emulsifier is included. Preferred emulsifiers are phytosterols. In preparing the solid form of the mix, the emulsifier and hard fat having a melting point of from about 29°C to about 66°C are formed together as a mixture and solidified. The emulsifier is then added to the melted fat and mixed until a clear solution is obtained.

40 [0009] U.S. Patent No. 3,865,939 is directed to edible cooking and salad oil compositions having enhanced hypocholesterolemic properties including plant sterols. The limited solubility of plant sterols in any solvent system is noted. A solubilizing agent to solubilize the plant sterol in the oil is selected from the group consisting of fatty acids, monoesters of fatty acids with polyhydric alcohols and alkanols. The invention is said also to contemplate peanut butter, mayonnaise, ice cream and margarine spreads. A solubilizing agent may be selected from a group including simple esters of fatty acids such as monoglycerides.

45 [0010] U.S. Patent No. 5,502,045 is directed to a beta-sitosterol fatty acid ester or mixture thereof which lowers cholesterol levels. In Example 5 a beta-sitosterol ester mixture is added to the fatty part of a conventional soft margarine.

50 [0011] Eng. U.S. Patent No. 4,195,984 is directed to a pharmaceutical preparation comprising a taste-stable aqueous suspension of tall oil sitosterols.

[0012] Beta sitosterol is described as being the most effective of the sterols for lowering serum cholesterol. Because

of certain physical properties of the sterols, it is said not to have been practical to provide a pharmaceutical suspension for oral administration which contains much more than 20 w/v of sitosterols. It is said that in order for sitosterols to be the most effective in lowering serum cholesterol the medicament must reach the gastrointestinal tract in a finely divided dispersed state.

[0013] Ong also reports that sitosterols do not lend themselves readily to incorporation into an aqueous preparation for oral administration that has a pleasant mouthfeel.

[0014] The Ong invention is directed to an aqueous pharmaceutical suspension comprised of finely divided tall oil sitosterols, a pharmaceutically acceptable chelating agent, sodium carboxymethylcellulose, sorbitol, a pharmaceutically acceptable surfactant, simethicone and water. The product is said to have an acceptable taste and mouthfeel that does not change over an extended storage period.

[0015] The pharmaceutical suspension of Ong may contain up to about 25 w/v of finely divided tall oil sitosterols. At about 20% tall oil sitosterols, the suspension is said not to be excessively viscous and is relatively easy to pour, having both good physical and chemical stability. A tall oil sitosterols preparation is said to develop no taste change after one year of shelf storage as compared with bitter taste development within two weeks at room temperature for a suspension of tall oil sitosterols not within the Ong invention. It is said that tall oil sitosterols are very hydrophobic and stubbornly resist wet wetting. Vigorous continuous agitation is said to be required to disperse the tall oil sitosterols in the vehicle.

[0016] The Ong phytosterol are said to be ground to a mean particle size of 25 microns or below by use of an air mill, high energy hammermill or air filtration mill under refrigeration or through the use of finely ground dry ice.

[0017] EP 259 636 discloses an emulsified or solubilized sterol composition wherein the sterols are emulsified or solubilized in an aqueous solution of polyhydroxy compounds containing sucrose fatty acid esters and/or polyglycerol fatty acid esters or liquid polyhydroxy compounds. A considerably high shelf stability is said to result and the invention is said to be extremely useful in various products including food, cosmetics, drugs and agricultural chemicals.

[0018] Sterols are described as high melting compounds which are hardly soluble in water and have a melting point of approximately 150°C. It is said to be difficult to obtain a stable, emulsified or solubilized composition. Betasitosterol is mentioned among the sterols which may be used in the '636 invention.

[0019] The compositions are prepared by, for example, adding sucrose ester and/or polyglycerol fatty acid ester to an aqueous solution of polyhydroxy compound, heating the mixture to 50 to 60°C, adding powdery sterols, stirring the obtained mixture at 50-90°C to dissolve the sterols and diluting the obtained solution if required. The compositions are said to show stable emulsification or solubilization without causing any separation of the sterols.

[0020] "Effect of Plant Sterols on Lipids and Atherosclerosis", Pollack, D.J., Pharmac. Ther., 31, 177-208 (1965) is reported in U.S. Patent No. 5,244,887 as suggesting the inclusion of plant sterols such as betasitosterol in such foods as butter and margarine to counteract not only the cholesterol in butter, but all other dietary cholesterol and cholesterol from non-dietary sources available for absorption and reabsorption.

#### Summary of the Invention

[0021] The present invention is directed to the discovery that phytosterols and other high melting lipids can be used to impart structure to water and fat continuous spreads and other products including aqueous phases. As mentioned above, U.S. Patent No. 3,885,939 discloses the well known difficulties in solubilizing plant sterols in any solvent system.

[0022] In the present invention, an aqueous dispersion of phytosterol or other high melting lipid is formed which is finely dispersed and stable, wherein the phytosterols or other high melting lipid serve to structure the aqueous dispersion. This is particularly useful in water and fat continuous spreads and other food systems or compositions, wherein the aqueous phase is structured by the phytosterol or other high melting lipid.

[0023] By using phytosterols or other high melting lipids as structuring agents, it is possible to avoid or minimize the use of saturated fat and other traditional structure-imparting ingredients in food products. For example, the presence of conventional thickeners such as gelatin and xanthan gum can be minimized or avoided. Moreover, even the inclusion of partially hydrogenated fat, which generally include trans fatty acids, can be avoided by use of phytosterols or other high melting lipids as structuring agents. At the same time, an added benefit where phytosterols are used is the reported cholesterol lowering effect of phytosterols. An even further benefit is the reduced calories which result from using high melting lipids as structuring agent since phytosterols and some other high melting lipids are barely or completely non-digestible.

[0024] Although a particularly advantageous use of the aqueous dispersions of the invention is in the preparation of water continuous low or no fat spreads, other food products can benefit from inclusion of the aqueous dispersions or suspensions according to the invention. These include fat continuous spreads which may be either vegetable oil or butter fat based, bi-continuous spreads, dressings, beverages, dairy products, such as milk, cheese, yogurt, non-dairy coffee whiteners, beverages, ice cream, and confections such as candy or chocolate.

[0025] The aqueous dispersions are useful as such in the preparation of foods and other products. Indeed, with the

dispersions and process of the invention, it is possible to make very concentrated phytosterol dispersions or suspensions, which have a number of important functional uses such as structuring, bodying and bulking agents, and whitening/opacity providers, especially in reduced and low fat foods.

[0026] The phytosterols and other high melting lipids are preferably present in the dispersion or suspension as very finely divided particles having a size of 15 microns or less, preferably 10 microns or less. The dispersion or suspension also include a non-phytosterol emulsifier. The inclusion of a non-sterol emulsifier may be omitted where sterols have been esterified with highly hydrophilic compounds such as citric acid, tartaric acid, for example. Such a chemical modification to phytosterols or other sterols would preclude the need to use a separate emulsifier of the type such as mono and diglyceride and polysorbate 60 in preparing the dispersion or suspension. The weight to weight ratio of emulsifier to phytosterol or other high melting lipid in the aqueous phase is less than 1:2, preferably less than 1:2.25, most preferably less than 1:3. Moreover, the aqueous phase dispersion or suspension according to the invention need not to include a large amount of triglyceride fat or other non-high melting lipid. However these non-high melting lipids can be present as well. If present the weight to weight ratio of non-phytosterol, non-high melting lipid to phytosterol or other high melting lipid in the aqueous phase is preferably less than 1:5, more preferably less than 1:8. Preferred phytosterols are beta sitosterol, campesterol, stigmasterol, brassicasterol and ergosterol.

[0027] A particularly advantageous use of the aqueous dispersions of the invention is in an oil-in-water-in-oil spread. In such spreads, the phytosterols or other high melting lipids are preferably used to structure both the continuous oil phase (external phase) and the dispersed aqueous phase. It has been found that such spreads including a continuous oil phase having phytosterols or other high melting lipids, a dispersed aqueous phase having phytosterols or other high melting lipids and a second oil phase dispersed in the aqueous phase have a reduced tendency toward oil separation and therefore an increased product stability. Preferably the continuous fat phase comprises from 0.5 to 5 wt. % phytosterol or other high melting lipid and the dispersed aqueous phase comprises from 2 to 15 wt. % phytosterol or other high melting lipid. The aqueous dispersions or suspensions of the invention are preferably prepared by melting the phytosterol (or other high melting lipid) and the emulsifier and dispersing the molten phytosterol (or other high melting lipid) and emulsifier in water under shear. While not wanting to be limited by theory, it is believed that the step of melting the high melting phytosterols with surfactant prior to dispersing in water with or without surfactant contributes importantly to the ability to prepare a very fine dispersion with the use of high shear mixing or homogenization of the phytosterol or other high melting lipid. Preferably the phytosterols or other high melting lipid in the present process and dispersions have been reduced to a size of 15 microns or lower, preferably 10 microns or lower.

#### Detailed Description of the Invention

[0028] The high melting lipid of the invention is preferably a phytosterol, i.e. plant sterols, such as alpha sitosterol, beta sitosterol, stigmasterol, ergosterol and campesterol, alpha spinosterol and brassicasterol. Although the foregoing are some of the more important phytosterols, at least 44 phytosterols have been identified and it will be apparent to one of ordinary skill that many of these will be appropriate for the present invention. Phytosterols are identified in bean (1993) phytosterols in "Advances in Lipid Research", pages 193-218, Paoletti, and Kirilchevsky, (Eds) Academic Press, NY, the disclosure of which is incorporated herein by reference. The disclosure of "Effect of Plant Sterols on Lipids and Atherosclerosis", Pollack, O.J., Pharmac. Ther., 31, 177-208 (1985) mentioned above is also incorporated by reference herein.

[0029] Many sources of phytosterols are known. Among sources are disclosed in Pollak "Effect of Plant Sterols on Serum Lipids and Atherosclerosis", Pharm.

[0030] Ther. Vol. 31, pp. 177-208, 1985, the disclosure of which is hereby incorporated by reference. See especially Table 7 on page 202. Among the more important sources are rice bran, corn bran, corn germ, wheat germ oil, corn oil, safflower oil, oat oil, olive oil, cotton seed oil, soybean oil, peanut oil, black tea, orange juice, Valencia, green tea, Colocasia, kale, broccoli, sesame seeds, shea oils, grapeseed oil, rapeseed oil, linseed oil, canola oil, tall oil from wood pulp and other resinous oil from wood pulp.

[0031] While particular benefit is obtained when the invention is used to emulsify or solubilize phytosterols or their esters, especially those which have been shown to have a cholesterol lowering benefit, zoosterols, fungal, algal and microbial sterols, and other high melting sterols and other lipids may also be used, as appropriate. Among the known zoosterols are cholesterol, 24-methylcholesterol, 7,22-dihydroxycholesterol and desmosterol.

[0032] It will generally be desirable to employ high purity and practical grade sterols and other high melting lipids which are suitable for ingestion by humans.

[0033] In addition to zoosterols, phytosterols and other sterols, it is believed that the present invention may be used advantageously with other high melting, water insoluble lipids. The high melting, water insoluble, sterols and other lipids of the invention have a melting point within the range of 75-200°C. Especially preferred are lipids with melting points of 100-200°C, and especially from 125-175°C.

[0034] Other classes of high melting lipids, in addition to the sterols, which may be used herein are the waxes in

particular, carnauba wax, bees wax, waxes and wax esters from vegetable oil sources, but also sterols, sterolesters, standols, stanolesters, hardened vegetable oils, saturated triglyceride fractions of vegetable oils, mono- and diglycerides, can be used. The phytosterols however are the preferred materials.

[0035] Melting point may be measured by known methods such as the AOCS capillary tube method and/or the Thomas-Hoover Uni-Melt melting point apparatus, ex. Thomas Scientific, Swedesboro, N.J.

[0036] The invention is used to greatest advantage when employing phytosterols and other sterols which have not been esterified. Phytosterols which have been esterified are more readily dissolved in oil phases and do not face to as great an extent the problem of solubilization and dispersion in food products having continuous fat phases. However, it may be appropriate under certain circumstances to utilize esterified phytosterols and other high melting lipids, so long as they are high melting, immiscible in water and fall in the desired melting temperature range of 75-200°C.

[0037] While the invention has been described as being particularly relevant to sterols, the invention may also be applied to their hydrogenated counterparts, such as phytosterols and to other chemically modified sterols. Chemical modifications include in addition to complete and partial hydrogenation, esterification, including interesterification. Examples of phytosterols include campestanol, 22,23 dihydrobrassicastanol, beta-sitostanol and clonastanol. Fatty acids esterified to the sterols include long and short chain fatty acids, i.e. C<sub>1</sub>-C<sub>22</sub>.

[0038] The phytosterols or other high melting lipids will suitably comprise about 1 to about 75% of the aqueous dispersions, preferably from 5 to 40 wt. %. However for other applications other amounts can also be used.

[0039] Many emulsifiers may be used to disperse the phytosterols or other high melting lipids. Preferred emulsifiers include polyglycerol esters and tweens, especially polysorbate 60. Other examples of emulsifiers which may be used include mono- and diglycerides, e.g., Myverol 18-04 available from Quest International, Hoffman Estates, Ill., sodium stearoyl lactylate, and polysorbates. Most preferred are oil-in-water emulsifiers.

[0040] The aqueous dispersions according to the invention may include, in addition to the phytosterols or other high melting lipids, ingredients such as the following: water, salt, flavors, preservatives, gums, starches, gelatin, milk and milk protein, colors, acidulants such as citric acid. Obviously, the aqueous dispersion may contain ingredients destined for the ultimate food product to be prepared from the dispersion.

[0041] The aqueous phytosterol and other high melting lipid dispersion of the invention may be concentrated by centrifugation, decantation, evaporation or other methods.

[0042] The concentration of the phytosterols or other high melting lipids in the aqueous dispersion can range from 0.1 to 99 wt. %, especially from 5 to 75, more particularly from 10 to 50.

[0043] As indicated above, a preferred method of preparing the aqueous dispersion involves mixing molten phytosterol or other high melting lipid together with molten surfactants in water. Preferably the surfactant level in the molten phytosterol phase is 1-20 wt. %. Alternatively, the molten surfactant can be incorporated separately into the aqueous phase.

[0044] A bicontinuous spread can suitably be made by dispersing a liquid oil into an aqueous dispersion of high melting lipid to form a first dispersion and then dispersing said first dispersion into a high melting lipid-containing oil phase. In that instance a product is made, wherein the continuous oil phase comprises 1-25 wt.% high melting lipids, the aqueous phase comprises 1-25 wt.% of one or more high melting lipid and the continuous oil phase comprises 25-95 wt. % of the spread and the internal oil phase comprises from 0 to 70 wt. % of the spread, the aqueous phase comprising 0 to 75 wt. % of the spread.

[0045] Among the foods in which the dispersions of the invention can provide structuring include water continuous spreads, fat continuous spreads, bicontinuous spreads, dressings, drinks, dairy products (such as milk, yogurt, cheese, cream cheese) dry mixes, powdered non-dairy coffee whiteners, milkshake mixes, confections, ice creams, instant milks, cake mixes and other food and pharmaceutical preparations.

[0046] As indicated above, the aqueous dispersions can be used to structure water continuous spreads or oil-in-water-in-oil spreads. In such case, the phytosterols preferably are present to structure both the continuous oil external phase and the dispersed aqueous phase.

[0047] Although not required, if desired the aqueous dispersions of the invention can be used in conjunction with other structuring agents in the spreads and other food products of the present invention. Such structuring agents include the mesomorphic phases of edible surfactant disclosed in WO 92/09209, the disclosure which is incorporated herein by reference.

[0048] Spreads according to the embodiment generally contain from less than 85% by weight of edible triglyceride materials. Suitable edible triglyceride materials are for example disclosed in *Bailey's Industrial Oil and Fat Products* (1979). In higher fat spreads, the level of triglyceride material will generally be more than 60% and less than 80%, preferably from 70 to 79% by weight. In spreads of reduced fat content the level of triglycerides will generally be from 30-60%, more generally from 35 to 45% by weight. In very low fat spreads the level of triglycerides will generally be from 0-40%, for example 30%, 25%, 20% or even 10% or about 0%. Other fatty materials, for example sucrose polyesters may be used as a replacement for part or all of the triglyceride material. Preferred water continuous spreads comprise 0-85 wt. % fat and 100-15 wt. % continuous aqueous phase. Preferred fat continuous spreads comprise 15-80

wt. % fat and 85-20 wt. % water phase.

[0049] The phytosterol or other high melting lipid material for use in spreads is preferably used at a level of from 5-50 wt. % of the aqueous phase, more preferred from 10-50%, most preferred from 20 to 40 % by weight. Spreads may comprise additional surfactants to those used to disperse the high melting lipid, for instance, monoglycerides and lecithins, ionic edible surfactant such as lactylated fatty acid salts and phosphatidic acid.

[0050] The water phase of the water continuous spread can suitably contain a non-phytosterol emulsifier and high melting lipid in a w/w-ratio of less than 1:2. The aqueous phase can also contain a non-high melting, non-phytosterol lipid. In that instance the w/w ratio of the non-phytosterol lipid to the high melting phytosterol is less than 1:6. The fat phase of fat continuous spreads can include structuring lipids, selected from high melting lipids and not high melting lipids. The high melting lipids preferably being phytosterols.

[0051] In addition to the above mentioned ingredients, spreads in accordance with the invention may optionally contain further ingredients suitable for use in spreads. Examples of these materials are gelling agents, thickening agents, sugars, eg sucrose or lactose, or other sweetener materials, EDTA, spices, salt, bulking agents, flavoring materials, coloring materials, proteins, acids etc. Suitable biopolymer materials which may be included in spreads include, for example, milk protein, gelatin, soy protein, xanthan gum, locust bean gum, hydrolyzed starches (for example PaselliSA2 and N-oil), and microcrystalline cellulose. Other gelling and thickening agents which may be used include but are not limited to carrageenan, pectin, gellan gum, agar, guar, alginate, maltodextrin, native and modified starches, and pre-gelatinized starches. Appropriate aqueous and fat phase ingredients are found in Cain et al. US Patent No. 4,917,915 and Norton et al. US Patent Nos. 5,194,285 and 5,151,290, the disclosures of which are hereby incorporated by reference.

[0052] Various sources for the gelling agents include plants, including marine plants, microorganisms, and animals. The amount of biopolymer, if any, in spreads of the invention is dependent on the desired degree of gelling and the presence of other ingredients in the composition. The amount of gelling agent may lie between 0 and 30%, mostly between 0.1 and 25% based on the weight of the aqueous phase of the spread. If hydrolyzed starches are present their level may be from 2-20%; other gelling agents may be used at levels of up to 10%, mostly 1-7%, most preferred 2-5% all percentages being based on the weight of the aqueous phase. Particularly preferred are combination of say 2-15% hydrolyzed starch and 0.5-5% of other gelling materials, especially gelling materials including gelatin.

[0053] In addition to or in combination with the above, the aqueous phase of spreads or other foods may include the following ingredients: dairy ingredients such as buttermilk, skim milk, milk, salt, acidulants, such as lactic acid and citric acid, butter, yogurt, whey, caseinate, milk proteins, vegetable proteins, vitamins and preservatives such as potassium sorbate and sodium benzoate.

[0054] The balance of the spread composition is generally water, which may be incorporated at levels of up to 99.9% by weight, more general from 10 to 98%, preferably from 20 to 97% by weight. Spreads according to the invention may be fat and/or water continuous.

[0055] Where the spread or other food product of the invention includes a fat phase, the composition of the fatty phase preferably comprises one or more vegetable oils, preferably sunflower oil, soybean oil, rapeseed oil, canola oil, corn oil, peanut/groundnut oil and the like. Although not generally preferred, if desired, dairy and other animal fat may also be used. Dairy, other animal fat sources and miscellaneous fat sources include milk (milk fat), buttermilk, fish oil, lard and tallow. If desired, the fat may be hydrogenated, fractionated and/or interesterified, but again it will usually be less desirable to include hydrogenated fat, which will be saturated and which may include trans fatty acids.

[0056] While the fat that is applied in these fat based food products can be any fat, such as dairy fat and/or vegetable fat, if fat is present, for health reasons the use of one or more vegetable fat sources is preferred. In particular, the use of liquid fats is preferred. The fat can be one single fat or a blend. The use of fat compositions comprising a considerable amount of PUFA (poly unsaturated fatty acid) rich triglycerides in addition to the use of the sterol/sterol ester mixture is in particular considered highly beneficial. For example, oils of sunflower, safflower, rapeseed, linseed, linola and/or soybean can be used in a preferred embodiment. Also the fat compositions mentioned in Netherlands patent documents no.

NL 143115, NL 175559, NL 155436, NL 149687, NL 155177, European patent documents EP 41303, EP 209176, EP 249282, and EP 470858, the disclosures of which are incorporated by reference herein, are highly suitable.

[0057] If a fat blend is used, it is preferred that it comprises at least 30%, and more preferred at least 45% of polyunsaturated fatty acid moieties, based on the total weight amount of the fat in the fat based food product. So a strong effect on the cholesterol lowering effect is obtained if use is made of an optimal ratio of sterol and sterol-esters as set forth in this application in a food product in which a fat blend comprising at least 30 wt. % of PUFA rich triglycerides is used.

[0058] Where butterfat is used for preparing spreads of the invention, or where the spreads are butter, it is preferred that the amount of phytosterol is in the range of 5-15%, preferably 10-15%. As the consumption of butter is considered less beneficial for consumers health, the present invention is in particular suitable for making butter or butter-melanges containing spreads, as the negative effect associated with the butter consumption can be minimized or even reversed.

[0059] Generally, dressings or mayonnaise are oil in water emulsions. The oil phase of the emulsion generally is 0 to 85% by weight of the product. For higher fat products the level of triglycerides is generally from 60-85%, especially from 65-80% by weight. For salad dressings the level of fat is generally from 10-60%, more preferred from 15 to 40%. Low or no-fat containing dressings may for example contain triglyceride levels of 0, 5, 10 or 15% by weight.

[0060] Other fatty materials such as for example polyol fatty acids ester may be used as a replacement for part or all of the triglyceride materials in the dressings or other foods of the invention.

[0061] The level of edible surfactant material in the dressing will generally be from 0.1 to 15%, more preferred from 1-10%, most preferred from 2 to 8% by weight. Preferably the level of nonionic edible surfactant is from 0.1 to 15%, more preferred, 0.5-10%, most preferred 1 to 8% by weight. Especially preferred are monoglycerides as nonionic edible surfactants. Preferably the level of ionic edible surfactant is from 0 to 5%, more preferred 0.05 to 2%, most preferred 0.1 to 0.5% by weight.

[0062] Dressings are in general low pH products with a preferred pH of from 2-6, more preferred 3-5, for example about 3.5. For the use in dressings the preferred anionic is the diacetyl tartaric ester of monoglycerides (in the examples Admul DATEM 1935 ex. Quest Int. has been used). Also an anionic phospholipid such as phosphatidic acid can be applied.

[0063] In addition to the above mentioned ingredients dressings in accordance with the present invention optionally may contain one or more other ingredients which may suitably be incorporated into dressings and/or mayonnaise. Examples of these materials are emulsifiers, for example egg-yolk or derivatives thereof, stabilizers, acidifiers, biopolymers, for example hydrolysed starches and/or gums or gelatin, bulking agents, flavors, coloring agents etc. The balance or the composition is water, which could advantageously be incorporated at levels of from 0.1-99.9%, more preferred 20-89%, most preferred 50 to 98% by weight.

[0064] The dispersions of the invention are useful as natural, non-caloric multifunctional ingredients in a wide range of food and pharmaceutical products. The aqueous phytosterol dispersions according to the invention can be used as hypocholesterolemic agents, as a non-caloric bulking agent, as a structuring and thickening material, as coloring, clouding and/or opacity ingredients, as a high melting carrier for flavors, colors and other materials in a broad spectrum of food and pharmaceutical preparations. The phytosterols or other high melting lipids can also be used as high melting encapsulation materials.

[0065] Owing to their structuring functionality, the phytosterols and other high melting lipids can be used to replace fat structuring methods such as hydrogenation, interesterification, and use of natural hard fats such as tropical oils and/or animal fats. The aqueous phytosterol or high melting lipid dispersions can be used to replace conventional water structuring agents, as well, such as proteins, carbohydrates, gelatins and other thickeners and stabilizers. Eliminating partially hydrogenated fats removes trans fatty acids and reduces saturated fatty acids and calories. Moreover, the elimination of hydrogenated fats reduces the perception that the products are somehow "unnatural."

[0066] Shear can be generated in preparation of the dispersion of the invention by using, eg a turbo mixer, a colloid mill, a ball mill, a homogenizer or other mechanical or sonic devices.

[0067] The particle size measurements may be performed by using a Coulter LS particle size analyzer, ex. Coulter, Miami, FL or by Particle Sizing Systems Inc. Models 770 Accusizer and Nicomp 370, Santa Barbara, CA.

[0068] Preferably the phytosterols or other high melting lipids have a particle size of 15 microns or lower. Preferably, at least 90% and more preferably 100% of particle sizes fall within a range of between 10 nanometers and 50 microns.

[0069] Materials which are typically used include phytosterols either pure or technical grade, either in stanol or stanol form; saturated distilled mono and diglycerides, e.g. Myerol 18-04; water; and polysorbate 60 (Tween 60).

[0070] Unless stated otherwise or required by context, the terms "fat" and "oil" are used interchangeable herein. Where a phase is said to constitute essentially the entire product, it is meant that such phase constitutes at least 98 wt. %, especially more than 99 wt. % of such product.

[0071] Where in this application phytosterols are mentioned, phytosterols, phytostanols, or mixtures thereof may be used as well. Likewise, where sterols are used in this application stanols are also contemplated. Thus, for instance, 4-desmethylsterols, 4-monomethylsterols and 4,4'-dimethylsterols, their stanol equivalents and mixtures thereof in any combination may all be useful.

[0072] Equipment which is typically employed includes Glass Beakers 250 ml and 2000 ml; hotplate and microwave oven; high shear turbomixer such as a Silverson with a fine screen gram scale, convention oven to melt phytosterol mix at about 150°C, and centrifuge.



Example 1

## Phytosterol Water Dispersion Process

[0073]

1. Melt 90g of phytosterols and 10g of Myverol 18-04 together in a 250 ml beaker.
2. Fill each of two 2000ml beakers with about 1500ml of water and heat to a boil in a microwave oven.
3. Place first 2000 ml beaker with the hot water on a hotplate set for about 95C which has been placed directly below the Turbomixer agitator shaft.
4. Turn Turbomixer on and gradually increase shaft speed until water is turbulent and a good vortex has formed.
5. Slowly add molten (150C) 90/10 phytosterol/18-04 mixture to vortex and increase Turbomixer speed to submerge and quickly disperse the molten mix which will have a tendency to foam and set up quickly on the water's surface. Caution: Addition of molten 90/10 mix must be controlled to keep foaming to a minimum and the temperature mix must not drop by more than a few degrees during this step. Reheat 90/10 mix as necessary and maintain temperature of water at 95C by use of the hot plate.
6. After dispersing the first half of the 90/10 mix (about 50g) in the first beaker with 1500 ml of hot water continue to mix the phytosterol dispersion for about 2 to 3 minutes at high speed. Remove the first beaker from the Turbomixer and begin dispersing the remaining 90/10 mix in the second beaker containing 1500ml of hot water. Follow the same procedures and precautions.
7. Allow phytosterol dispersion to separate. Decant or siphon off water layer. Taste phytosterol layer; if bitterness exist combine the two phytosterol layers into one beaker and water wash five times using hot water and Turbomixer for each wash cycle. For each cycle, decant wash water and use about 1500 ml of fresh hot water.
8. The washed or nonwashed phytosterol dispersion is concentrated in a centrifuge to a moisture level of about 70 to 75%. Store the concentrated phytosterol dispersion refrigerated.
9. A similar 90/10 phytosterol dispersion can be made by substituting 10g of polysorbate 60 for 10g of Myverol 18-04. Same procedure and precautions apply.
10. The dispersion process (particle size reduction) is facilitated by using 1% polysorbate 60 in the hot water to which the molten phytosterols are added under Turbomix agitation. However, use of polysorbate 60 in the water phase makes separation of the phytosterol layer more difficult and time consuming.

## Typical Formulations Based on Water Dispersed Phytosterols:

[0074] In all examples the phytosterol applied (Phyto) was derived from soybean and consisted primarily of Beta-sitosterol (45%), campesterol (27%), stigmasterol (21%) and some minor other sterols (total 7%).

Example 2

[0075] 65% Oil w/o spread with 10% phytosterols and no trans fats

Part I-Beaker with Oil Phase	
Sunflower Oil	269g
100% Phytosterols-pure technical	10g
	270g @ 150C

Part II-Beaker with Water Phase	
90/10 Phyto/18-04 Water Dispersion (72.5% Moisture)	122g @ 20C
Salt/Flavor/color	8g
	400g Cool to 30C and fill

[0076] Procedure: Add Part I to Part II under Turbomixer agitation. Cool w/o emulsion in a cold water bath (larger beaker) while emulsion is under agitation. Cool down to about 30C and fill in to cup. Refrigerate.

Example 3

[0077] 55% Oil Duplex Emulsion Spread with 10% Phytosterols &amp; no trans fats

Part I - Beaker with o/w Emulsion	
Sunflower Oil	70g @ 20/25C.
90/10 Phyto/18-04 Water Dispersion	121g @ 20C.
Salt	8g
Polysorbate 60	1g
	200g @ 20/25C.

Part II - Beaker with External Oil Phase	
Sunflower Oil	188g @ 150C.
Myverol 18-04	2g @ 150C.
100% Phytosterols - pure/technical	10g @ 150C.
Flavor/Color	qs add *
	400g

\* add flavor/color when Part I &amp; Part II are being combined

[0078] Procedure: Using Turbomixer, prepare Part I by dispersing SF Oil in the 90/10 Phyto/18-04 dispersion in which the Polysorbate 60 has been well incorporated. Then add the Part I o/w emulsion to the beaker containing Part II again by using the Turbomixer. Use a cold water bath to cool this fat continuous emulsion down to about 30C. Fill into cups. Refrigerate.

Example 4

[0079] Formulations for 0%, 6% and 24% fat water continuous spreads with 10% phytosterols:

Ingredients	0%	6%	24%
90/10 PHYTO/18-04 (72.5% moisture)	162	162	162
SF oil	---	24	96
Water (95C)	215.95	193.15	122.45
Gelatin (beef)	5	4.5	4
Starch, Remyline AP	5	4.5	4
Lactose	4	3.8	3.5
Lactic Acid	.3	.3	.3
K Sorbate	.05	.05	.05
Salt	6.5	6.5	6.5
Buttermilk Powder	1	1	1
Beta Carotene CWS 1 %	.15	.15	.15
Flavor and Vitamins	.05	.05	.05
	400g	400g	400g

[0080] Preparation: Disperse gelatin and starch in the 90C water using the turbomixer. Then add remaining ingredients under agitation. Add flavors/vitamins and the 90/10 Phyto/18-04 dispersion last. Cool down to 20-25C pour in cups. Refrigerate.

Example 5

[0081] 0% fat reduced calorie mayonnaise



	Control	New
Titanium Dioxide	1.0	---
Vinegar 120 grain	4.5	4.5
Mustard Flour	0.5	0.5
Food Starch Modified	10.0	5.0
K Sorbate	0.1	0.1
Na Benzoate	0.1	0.1
Salt	2.0	2.0
Sugar	8.0	8.0
Beta Carotene	0.1	0.1
Natural Spice Flavor	0.2	0.2
Natural Egg Flavor	0.2	0.2
Phosphoric Acid	0.2	0.2
Water	73.1	39.1
90/10 PHYTO/18-04 @ 72.5% Moisture	---	40.0
	100.0	100.0

[0082] Preparation: Cook all ingredients other than the 90/10 Phyto/18-04 dispersion in starch cooker. Cool the cooked starch and add the phyto/sterol dispersion under mild agitation. Mill final mixture through a colloid mill.

[0083] Formula replaces modified food starch by 50% and use of the artificial food color titanium dioxide (whitening and opacity agent by 100%)

#### Example B

#### [0084] Creamy Italian Dressing

	Control	New
Soybean Oil	45.0	35.0
Water	40.4	11.0
Sugar	4.5	4.5
Vinegar 120 Grain	9.0	3.0
HVF Algin	0.2	0.1
Buttermilk Powder	2.2	1.0
Lactic Acid	0.3	0.3
Lemon Juice Cone	0.4	0.4
Salt	2.5	2.5
Minced Onion	0.2	0.2
Minced Garlic	0.5	0.5
Xanthan Gum	0.2	0.1
Red Bell Peppers	0.2	0.2
MSG	0.1	0.1
Spices	0.1	0.1
Polysorbate 60	0.2	---
90/10 PHYTO/18-04 Moisture 72.5%	---	40.0
	100.0	100.0

[0085] Preparation. Combine ingredients under agitation and process through colloid mill.

[0086] Formula eliminates use of polysorbate 60 and reduces use of food gums by 50%, buttermilk powder use by 50% and soybean oil usage by 20%.

[0087] It will be apparent that for commercialization the previously mentioned process steps would be upscaled to the appropriate process and equipment sizes, types and standards practiced in the particular or relevant food industry.

[0088] It should be understood of course that the specific forms of the invention herein illustrated and described are

intended to be representative only as certain changes may be made therein without departing from the clear teachings of the disclosure. Accordingly, reference should be made to the following appended claims in determining the full scope of the invention.

## Claims

1. An aqueous phase dispersion or suspension comprising
  - a) one or more high melting lipids having a mean size of 15 microns or lower, and
  - b) a non-sterol emulsifier, the w/w ratio of emulsifier to high melting lipid in said aqueous phase being less than 1:2.
2. The dispersion according to claim 1 wherein the high melting lipids have a mean size of 10 microns or less.
3. The dispersion according to claim 1-2, wherein the high melting lipids have a melting point within the range of 75-200°C.
4. The dispersion according to claims 1-3 wherein the high melting lipids are selected from the group consisting of: phytosterols, phytosterolesters, sterols, sterolesters, stanols, stanolesters, waxesters, hardened vegetable oils, saturated triglyceride fractions of vegetable oils, mono- and diglycerides.
5. The dispersion according to claims 1-4, comprising 0.1 - 99 wt % preferably 5-75 wt.%, more preferably 10-50 wt. % of the high melting lipids.
6. The dispersion according to claims 1-5 incorporated into a foodstuff selected from the group consisting of water-continuous spreads, fat continuous spreads, bicontinuous spreads, dressings, beverages, dairy products, milk, cheese, yogurt, non-dairy coffee whiteners, beverages, confections and ice cream.
7. The dispersion according to claims 1-5 wherein the emulsifier is selected from the group consisting of monoglycerides, diglycerides, polysorbates, sodium stearyl lactylate and polyglycerol esters.
8. The dispersion according to claims 1-5 wherein the emulsifier is an oil-in-water emulsifier.
9. The dispersion according to claims 1-5 wherein the w/w ratio of emulsifier to high melting lipid is less than 1:2.25, in particular less than 1:3.
10. The dispersion according to claims 1-5 wherein the high melting lipid is a phytosterol which is selected from the group consisting of Beta sitosterol, campesterol, stigmasterol, brassicasterol and ergosterol.
11. An aqueous phase dispersion or suspension according to claims 1-5, also comprising a non-high melting lipid in a w/w-ratio to high melting lipid of less than 1:3, preferably less than 1:5.
12. A water continuous edible spread according to claim 6 comprising a discontinuous fat phase constituting from 0 to 85 wt. % of said spread, and a continuous aqueous phase constituting from 15 to 100 wt. % of said spread.
13. The spread according to claim 12 wherein the aqueous phase comprises a non-phytosterol emulsifier, in a w/w ratio of said emulsifier to high melting lipid of less than 1:2.
14. The spread according to claims 12-13 wherein
  - a) the high melting lipids are phytosterols and have a mean size of 15 microns or lower, and
  - b) wherein the aqueous phase comprises a non-phytosterol emulsifier, and a non-high melting, non-phytosterol lipid, the w/w ratio of the non-phytosterol lipid, to the high melting phytosterol being less than 1:5
15. The spread according to claims 11-14 wherein the fat phase ranges from 0 to 40 wt. % of said spread, and wherein the aqueous phase constitutes from 60 to 100 wt. % of the spread.

16. The water continuous spread of claims 11-15 wherein the aqueous phase includes from 5 to 50 wt. % high melting lipids.
17. An edible, fat continuous spread according to claim 6 comprising a continuous fat phase constituting from 15 to 80 wt. % of said spread and a discontinuous aqueous phase constituting from 20 to 85 wt. % of said spread.
18. The fat continuous spread according to claim 17 wherein the fat phase includes a structuring lipid selected from the group consisting of a) high melting lipids and b) lipids which are not high melting
19. The fat continuous spread according to claims 17-18 wherein the structuring lipid is selected from the group consisting of hydrogenated oil, or interesterified oil, or fractionated oils, or non-hydrogenated, non-interesterified non-fractionated hardstock or hydrogenated hardstock fat, or interesterified hardstock fat, or hydrogenated hardstock fat.
20. The fat continuous spread according to claims 17-19 wherein the aqueous phase includes from 5 to 50 wt. % phytosterols.
21. The fat continuous spread according to claims 17-20 wherein the fat phase includes a structuring agent selected from the group consisting of hydrogenated and/or esterified phytosterols, free phytosterols, waxes in particular waxes from aliphatic long chain fatty acids.
22. The fat continuous spread according to claims 17-21 wherein the aqueous phase includes dispersed therein a further fat phase.
23. The fat continuous spread according to claims 17-22 wherein the continuous fat phase comprises phytosterols.
24. The fat continuous spread according to claims 17-23 wherein the continuous fat phase comprises from 0.5 to 6 wt. % high melting lipid based on the weight of the product, and the aqueous phase comprises from 2 to 15 wt. % high melting lipid based on the weight of the product.
25. A method for preparing an aqueous dispersion or suspension of high melting lipid comprising mixing together a molten high melting lipid, molten surfactant, and water under shear.
26. The method according to claim 25 wherein said shear is provided by a turbo mixer, a colloid mill, a ball mill or a homogenizer.
27. The method according to claims 25-26 further comprising concentrating the suspension or dispersion.
28. A process for making a spread comprising dispersing a liquid oil into an aqueous dispersion of high melting lipid to form a first dispersion and then dispersing said first dispersion into a high melting lipid-containing oil phase.
29. The process according to claim 28 wherein the continuous oil phase comprises 1-25 wt. % high melting lipids, the aqueous phase comprises 1-25 wt. % of one or more high melting lipid and the continuous oil phase comprises 25-95 wt. % of the spread and the internal oil phase comprises from 0 to 70 wt. % of the spread, the aqueous phase comprising 0 to 75 wt. % of the spread.
30. The dispersion according to claim 4 wherein the high melting lipid is a chemically modified sterol.
31. The dispersion according to claim 30 wherein the chemically modified sterol is 8 sitosterol.
32. A fat based food product comprising an aqueous dispersion or suspension including:
  - a) one or more high melting lipids having a mean size of 15 microns or lower and
  - b) a non-sterol emulsifier, the w/w ratio of emulsifier to high melting lipid in said aqueous phase being less than 1:2,

wherein the fat used in the product is a fat comprising at least 30 wt. % preferably at least 45 wt. % of PUFA rich triglycerides, calculated on the total weight of the fat present in the product.

33. A fat based food product comprising an aqueous dispersion or suspension including:

- a) one or more high melting lipids having a mean size of 15 microns or lower and
- b) a non-sterol emulsifier, the w/w ratio of emulsifier to high melting lipid in said aqueous phase being less than 1:2,

wherein the fat in the food product comprises butterfat, and the total amount of phytosterol is in the range of 5-15 wt. %

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Application Number  
EP 02 07 5153

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